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SAMPLING AND ANALYSIS PLAN DATA GAPS ASSESSMENT FOR TANK FARM 3 CATEGORY 1 AREAS NS NEWPORT RI

5/1/2013

TETRA TECH INC

SAP Worksheet #1 -- Title Page
(UFP-QAPP Manual Section 2.1)

SAMPLING AND ANALYSIS PLAN
(FIELD SAMPLING PLAN AND QUALITY ASSURANCE PROJECT PLAN)
MAY 2013

DATA GAPS ASSESSMENT
TANK FARM 3, CATEGORY 1 AREAS
NAVAL STATION NEWPORT
NEWPORT, RHODE ISLAND

PREPARED FOR:
NAVAL FACILITIES ENGINEERING COMMAND MID-ATLANTIC
9742 MARYLAND AVENUE
NORFOLK, VIRGINIA 23511-3095

PREPARED BY:
TETRA TECH
234 MALL BOULEVARD SUITE 260
KING OF PRUSSIA, PENNSYLVANIA 19046-1433
(610) 491-9688

PREPARED UNDER:
CONTRACT NUMBER N62470-08-D-1001
“CLEAN” CONTRACT TASK ORDER NO. WE59

SAP Worksheet #1 – Approval Page
(UFP-QAPP Manual Section 2.1)

Document Title: Draft Sampling and Analysis Plan, (Field Sampling Plan and Quality Assurance Project Plan), April 2011, Data Gaps Assessment, Tank Farm 3, Category 1 Areas, Naval Station Newport, Newport, Rhode Island

Lead Organization: Naval Facilities Engineering Command Mid-Atlantic

Preparer's Name and Organizational Affiliation: Tetra Tech

Preparer's Address and Telephone Number: 234 Mall Boulevard Suite 260, King of Prussia, Pennsylvania 19046-1433

Preparation Date (Day/Month/Year): April 2011

Investigative Organization's Project Manager: _____
Signature/Date
Dabra Seiken, CG, Tetra Tech

Investigative Organization's Project QA Manager: _____ 4-22-11
Signature/Date
Tom Johnston, PhD, Tetra Tech

Lead Organization's Project Manager: _____
Signature/Date
Roberto Pagtalunan, PE, NAVFAC Mid Atlantic

Lead Organization QA Officer: _____
Signature/Date
NAVFAC Chemist, NAVFAC Atlantic

Approval Signatures: _____
Signature/Date
Kymberlee Keckler, U.S. EPA

_____
Signature/Date
Gary Jablonski, RIDEM

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Tom Johnston, PhD, Tetra Tech

Lead Organization's Project Manager:

Signature/Date
Roberto Pagtalunan, PE, NAVFAC Mid Atlantic

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DN: c=US, o=U.S. Government, ou=DoD, ou=PKI, ou=USN, cn=NIELSEN.JANICE.L.1069943540
Date: 2011.04.28 14:14:20 -04'00'

Lead Organization QA Officer:

Signature/Date
NAVFAC Chemist, NAVFAC Atlantic

Approval Signatures:

Signature/Date
Kymberlee Keckler, U.S. EPA

Signature/Date
Gary Jablonski, RIDEM

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Preparer's Address and Telephone Number: 234 Mall Boulevard Suite 260, King of Prussia, Pennsylvania 19046-1433

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Investigative Organization's Project QA Manager:

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Tom Johnston, PhD, Tetra Tech

Lead Organization's Project Manager:

Signature/Date
Roberto Pagtalunan, PE, NAVFAC Mid Atlantic

Lead Organization QA Officer:

Signature/Date
NAVFAC Chemist, NAVFAC Atlantic

Approval Signatures:

Signature/Date
Kymberlee Keckler, U.S. EPA

Signature/Date
Pamela Crump, RIDEM

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Tom Johnston, PhD, Tetra Tech

Lead Organization's Project Manager:

Signature/Date
Roberto Pagtalunan, PE, NAVFAC Mid Atlantic

Lead Organization QA Officer:

Signature/Date
NAVFAC Chemist, NAVFAC Atlantic

Approval Signatures:

23 MAY 13

Signature/Date
Kymberlee Keckler, U.S. EPA

Signature/Date
Pamela Crump, RIDEM

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Preparation Date (Day/Month/Year): May 2013

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Dabra Seiken, CG, Tetra Tech

Investigative Organization's Project QA Manager:

Signature/Date
Tom Johnston, PhD, Tetra Tech

Lead Organization's Project Manager:

Signature/Date
Roberto Pagtalunan, PE, NAVFAC Mid Atlantic

Lead Organization QA Officer:

Signature/Date
NAVFAC Chemist, NAVFAC Atlantic

Approval Signatures:

Signature/Date
Kymberlee Keckler, U.S. EPA

PCL CL 5/23/13

Signature/Date
Pamela Crump, RIDEM

EXECUTIVE SUMMARY

This Sampling and Analysis Plan (SAP) presents the methodologies to be used for collecting data that will be utilized for determining the nature and extent of contamination related to past activities that have resulted in what are considered releases under the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) at Tank Farm 3 (Site 11), which is part of Naval Station (NAVSTA) Newport (formerly the Naval Education and Training Center [NETC] Newport). The data collected is also expected to be used to estimate whether risks from exposure of human and ecological receptors to site contaminants merit actions to further investigate or mitigate the risks, in an effort to protect human health and the environment.

NAVSTA Newport is located in the Towns of Newport, Middletown, and Portsmouth, Rhode Island, approximately 25 miles southeast of Providence. Tank Farm 3 (the Site) is situated at the southwestern portion of Portsmouth, Rhode Island, near the eastern shore of Narragansett Bay (Figure 1).

The Site is approximately 40 acres in size and is bordered by the Navy's Defense Highway to the northwest; Raytheon's Submarine Signal Division plant to the northeast, Bayview Estates (residential condominiums) to the southeast, and newer residential properties to the southwest (Figure 2). The Site consists of an upland area in the south central portion and a wetland area located along the eastern/northeastern boundary of the Site.

The Site is occupied by seven underground storage tanks (USTs) formerly used for bulk storage of petroleum products and the support buildings/structures/piping associated with the petroleum storage and distribution facility. The Site was used for the storage and distribution of petroleum products from the early 1940s until 1998.

The Defense Energy Support Center (DESC) operated the Site between 1974 until the 1990s, when the USTs were emptied and cleaned. Environmental investigations and remediation at the Site have previously occurred under the Rhode Island Department of Environmental Management (RIDEM) site remediation regulations by entities under contract to the DESC because the nature of the contaminants were primarily petroleum. The Navy has evaluated the previous data collected at the Site to determine the path forward to eventual site closure.

In accordance with decisions made by the Project Team, the Site has been broken up into Category 1 (CERCLA-regulated) areas and Category 2 (RIDEM UST Division regulated) areas. Also, RIDEM has identified several other areas of potential concern for which the scope of investigation has not been determined. The Category 3 areas will be further evaluated to determine if additional investigation of these areas, if any, will be performed as a Category 1 or Category 2 area.

This SAP addresses only Category 1 Areas. Based on a review of historical environmental investigations and remediation performed at the Site, data gaps for three Category 1 areas were identified. The Category 1 areas that will be further characterized under this SAP are:

- a former sand filter/burning chamber, also known as Area of Concern (AOC) 001;
- an electrical transformer area; also known as AOC 020, and
- an electrical control house (ECH), also known as Building 227.

Tank bottom sludge was previously deposited and burned without controls within the former sand filter/burn chamber (AOC 001). This sand filter was also used to collect groundwater from around the USTs. The sand filter has been cleaned and investigated/remediated with respect to environmental impacts by DESC. However, those efforts focused on contamination by total petroleum hydrocarbons (TPH). Following remediation of TPH-impacted soil, it was concluded that some TPH remains in two locations around the concrete sand filter structure.

Based on this information, additional data is necessary with respect to delineation of CERCLA contamination of soil near the sand filter, groundwater in the vicinity of the sand filter, and sediment in the area where the sand filter appears to have discharged to the wetlands around Lawton Brook. Soil and sediment will be sampled and analyzed for volatile organic compounds (VOCs), polynuclear aromatic hydrocarbons (PAHs), metals, and dioxins and furans (dioxins). Additionally, at the request of RIDEM, soil and sediment samples will be analyzed for TPH (gasoline-range organics [GRO] and extractable TPH [ExTPH]). Groundwater will be sampled and analyzed for VOCs, PAHs, and metals.

The electrical transformer area (AOC 020) is an area where a former transformer blockhouse was once present, and replaced with two pad-mounted electrical transformers. Previous environmental investigation in this area indicated the presence of polychlorinated biphenyls (PCBs) in soil. Based on the use of this area for electrical equipment and the presence of PCBs, additional sampling and analysis will be performed in this area to characterize the nature and extent of PCB contamination in soil and to confirm previous results that did not detect PCBs in groundwater.

The ECH (Building 227) houses electrical equipment, including a transformer and presumably batteries, for the former operation of the tank farm. The routine operation and maintenance of electrical equipment in this building could have resulted in a release to the environment of PCBs (from transformer oil) and/or metals (from batteries). Previous environmental investigations in this area were primarily focused on analyzing soil samples for TPH. Due to the nature of potential for releases as this location, additional soil sampling and analysis will be performed to determine if a release to the environment occurred, and to characterize the nature and extent of that contamination, if present. The analyte list for soil includes PCBs and metals and at the request of RIDEM, soil samples will also be analyzed for TPH (GRO and ExTPH). Groundwater sampling and analysis will also be performed to determine if a potential release has impacted groundwater quality.

Following completion of the investigation, the Navy will prepare a Data Gaps Report. This document will summarize the investigation activities, describe any issues encountered in the field along with corrective actions taken, provide tables comparing soil, sediment and groundwater sample results to screening levels, and provide figures depicting the locations sampled and the spatial distribution of contaminants. If concentrations of contaminants found indicate a potential for risk as described in this SAP, the project team will convene and determine the next steps.

SAP Worksheets

TABLES

NUMBER

FIGURES

NUMBER

FIGURES (cont.)

NUMBER

REFERENCES

APPENDICES

Acronyms

AOCs	Areas of Concern
BFB	bromofluorobenzene
bgs	Below Ground Surface
BTAG	Biological Technical Assistance Group
°C	Celsius
CCV	Continuing Calibration Verification
CDD	Chlorinated Dibenzo-p-Dioxins
CDF	Chlorinated Dibenzofurans
CERCLA	Comprehensive Environmental Response Compensation and Liability Act
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-Term Environmental Action Navy
CLP	Contract Laboratory Program
COD	Coefficient of Determination
CSM	Conceptual Site Model
CTO	Contract Task Order
DEC	Direct Exposure Criteria
DESC	Defense Energy Support Center
DGA	Data Gaps Assessment
DL	Detection Limit
DO	Dissolved Oxygen
DoD	Department of Defense
DQI	Data Quality Indicators
DQO	Data Quality Objective
DRO	Diesel Range Organic
DVM	Data Validation Manager
ECD	Electron Capture Detector
ECH	Electrical Control House
EDB	1,2-Dibromoethane
EDD	Electronic Data Deliverable
EDL	Estimated Detection Limit
EPA	United States Environmental Protection Agency
ExTPH	Extractable Total Petroleum Hydrocarbon
FID	Flame Ionization Detector
FOL	Field Operations Leader
g	Grams
GC	Gas Chromatograph
GIS	Geographic Information System
GPR	Ground Penetrating Radar
GRO	Gasoline Range Organics
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HCl	Hydrochloric Acid
HQ	Hazard Quotient
HSM	Health and Safety Manager
IAS	Initial Assessment Study
ICAL	Initial Calibration
ICS	Interference Check Sample
ICV	Initial Calibration Verification
IDW	Investigation-Derived Waste
IR	Installation Restoration
JP	jet propulsion
Katahdin	Katahdin Analytical Services
L	Liter
LCS	Laboratory Control Sample
LOD	Limits of Detection
LOQ	Limits of Quantitation

MCL	Maximum Contaminant Level
MEK	2-Butanone
mg/kg	Milligrams per Kilogram
mg/L	Milligrams per Liter
MIBK	4-Methyl-2-pentanone
mL	Milliliters
MLW	Mean Low Water
MPC	Measurement Performance Criteria
MS	Mass Spectrometer
MS	Matrix Spike
MSD	Matrix Spike Duplicate
NA	Not Applicable
NAD	North American Datum
NAVFAC	Naval Facilities Engineering Command
NAVSTA	Naval Station Newport
NETC	Naval Education and Training Center
NGVD	National Geodetic Vertical Datum
NIRIS	Naval Installation Restoration Information Solutions
NOAA	National Organic Atmospheric Administration
ORP	Oxygen Reduction Potential
OSHA	Occupational Safety and Health Administration
OWS	oil-water separators
oz	Ounce
PAH	Polynuclear Aromatic Hydrocarbon
PCB	Polychlorinated Biphenyl
PID	Photo Ionization Detector
pg/g	Picogram per gram
PM	Project Manager
PPE	Personal Protective Equipment
ppm	parts per million
PQO	Project Quality Objective
PSL	Project Screening Level
QA	Quality Assurance
QAM	Quality Assurance Manager
QC	Quality Control
RDEC	Residential Direct Exposure Criteria
RF	Response Factors
RI	Remedial Investigation
RIDEM	Rhode Island Department of Environmental Management
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RRT	Relative Retention Time
RSD	Relative Standard Deviation
RSL	Regional Screening Level
RT	Retention Time
RTC	Response to Comment
SAP	Sampling and Analysis Plan
SASE	Study Area Screening Evaluation
SCV	Secondary Chronic Levels
SDG	Sample Delivery Group
SIM	Selected Ion Monitoring
SIRAR	Site Investigation and Remedial Action Report
SL	Screening Levels
SOP	Standard Operating Procedure
SPCC	System Performance Check Compound
SQB	Sediment Quality Benchmarks
SQL	Structured Query Language
SQuiRT	Screening Quick Reference Tables
SSL	Soil Screening Level

SSO	Site Safety Officer
SVOC	Semi-Volatile Organic Compound
TBD	To-Be Determined
TEF	Toxicity Equivalency Factor
TEQ	Total Toxicity Equivalency
TPH	Total Petroleum Hydrocarbon
TVOC	Total Volatile Organics
TVPH	Total Volatile Petroleum Hydrocarbons
UCL	Upper Confidence Limit
UFP-QAPP	Uniform Federal Policy for Quality Assurance Plans
USEPA	U.S. Environmental Protection Agency
UST	Underground Storage Tank
VI	Vapor Intrusion
VISL	Vapor Intrusion Screening Level
VOC	Volatile Organic Compound
WHO	World Health Organization
.PDF	Portable Document Format
µg/kg	micrograms per kilogram

SAP Worksheet #2 – Sampling and Analysis Plan (SAP) Identifying Information

Site Name/Number: **Tank Farm 3, Naval Station (NAVSTA) Newport**
Operable Unit:
Contractor Name: **Tetra Tech**
Contract Number: **N62470-08-D-1001**
Contract Title: Naval Facilities Engineering Command (NAVFAC) Mid-Atlantic Comprehensive Long-Term Environmental Action Navy (CLEAN)
Work Assignment Number (optional): Contract Task Order (CTO) WE59

1. This SAP was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Plans (UFP-QAPP)* (U.S. Environmental Protection Agency [USEPA], 2005) and *EPA Guidance for Quality Assurance Project Plans, EPA Quality Assurance (QA)/G-5, Quality Assurance Manager (QAMS) (U.S. EPA 2002a)*.

2. Identify regulatory program: Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA).

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

	Date
Introductory Session - Tetra Tech, & Mid Atlantic	10/21/2010
Introductory Session (Remedial Project Manager [RPM] Meeting) – Tetra Tech, Mid Atlantic, USEPA Region I, Rhode Island Department of Environmental Management (RIDEM)	11/17/2010
Technical Session (Tetra Tech)	03/16/2011

5. List dates and titles of any SAP documents written for previous site work that are relevant to the current investigation.

Title	Date
Work Plan for Site Closure, Tank Farm 3, Foster Wheeler Environmental Corporation.	August 2002

6. List organizational partners (stakeholders) and connection with lead organization:

USEPA, Regulatory Oversight	NAVFAC, Mid-Atlantic – Responsible Party
RIDEM, Regulatory Oversight	Tetra Tech, Contractor to NAVFAC
NAVSTA, Property Holder	

7. Lead organization

U.S. Navy (Navy) (NAVFAC Mid Atlantic)

8. If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:

None

SAP Worksheet #3 -- Distribution List
(UFP-QAPP Manual Section 2.3.1)

Name of SAP Recipients	Title/Role	Organization	Telephone Number (Optional)	E-mail Address or Mailing Address	Document Control Number (Optional)
Roberto Pagtalunan, PE	RPM	NAVFAC Mid Atlantic	757-341-2010	roberto.pagtalunan@navy.mil	Not applicable (NA)
Deborah Moore	Installation Restoration (IR) Program Contact	NAVFAC Newport	401-841-1790	deborah.j.moore@navy.mil	NA
Dave Barclift	Ecological Risk Assessor	NAVFAC	215-897-4913	david.barclift@navy.mil	NA
Kymberlee Keckler	Remedial Project Manager (RPM)	USEPA Region 1 Federal Facilities	617-918-1385	keckler. kymberlee@epa.gov	NA
Pamela Crump	RPM	RIDEM Div Site Remediation	401-222-2797	pamela.crump@dem.ri.gov	NA
Dabra Seiken	Project Manager (PM)	Tetra Tech	978-474-8400	dabra.seiken@tetratech.com	NA
Tom Johnston (electronic copy)	QAM	Tetra Tech	412-921-8615	tom.johnston@tetratech.com	NA
Robin Clark	Field Operations Leader (FOL)/Project Geologist/ Site Safety Officer (SSO)	Tetra Tech	978-474-8400	robin.clark@ tetratech.com	NA
Kelly Carper	Project Chemist	Tetra Tech	412-921-7090	kelly.carper@tetratech.com	NA
Kelly Perkins	Laboratory PM	Katahdin Analytical Services (Katahdin)	207-874-2400	kperkins@katahdinlab.com	NA
Nilo Ligi	Laboratory PM	TestAmerica – West Sacramento (TestAmerica)	916-374-4427	Nilo.Ligi@testamericainc.com	NA
Glenn Wagner	Administrative Record Manager	Tetra Tech	412-320-2211	glenn.wagner@tetratech.com	NA

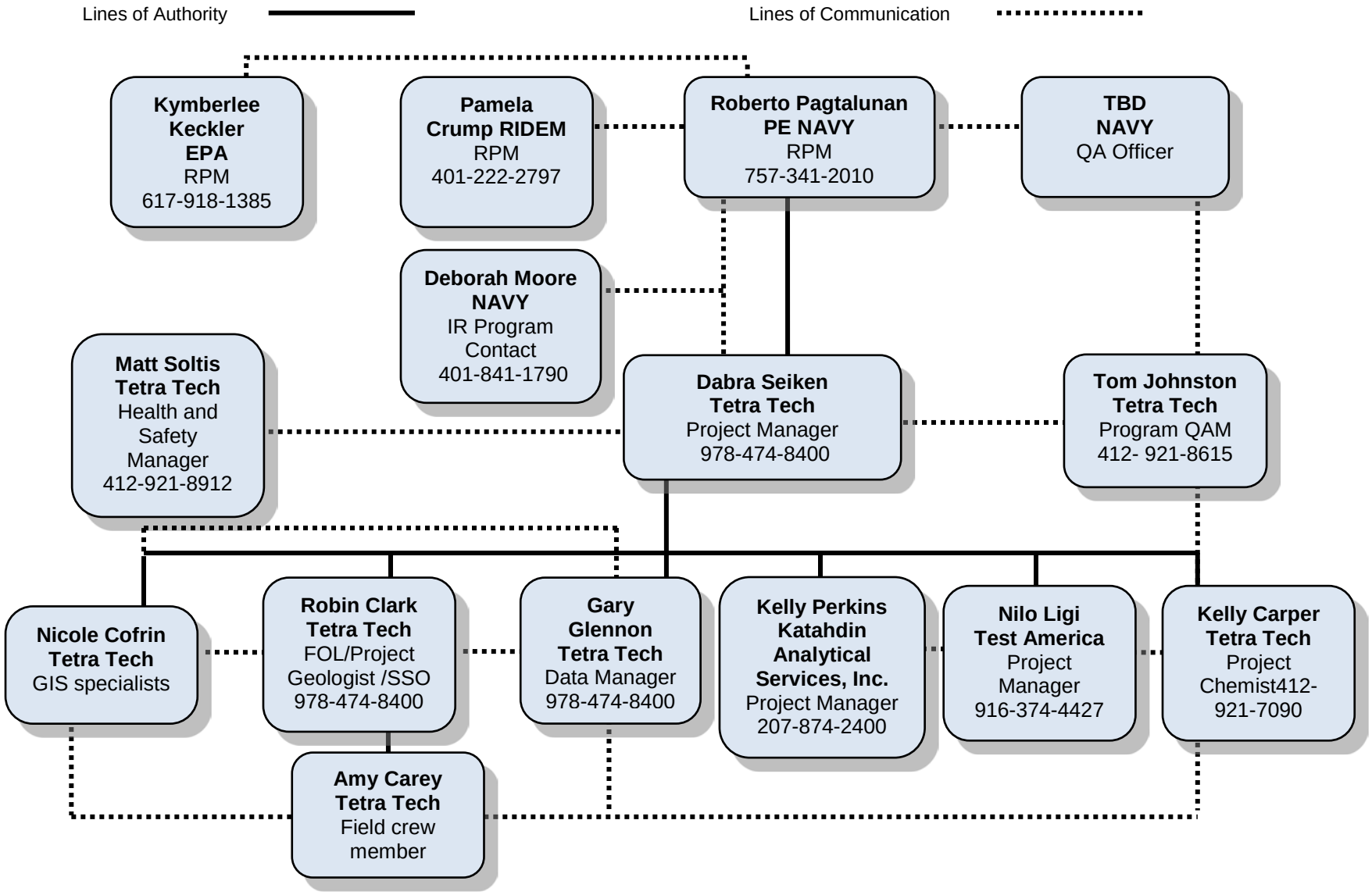
SAP Worksheet #4 -- Project Personnel Sign-Off Sheet
(UFP-QAPP Manual Section 2.3.2)

Project personnel who are responsible for implementing portions of the SAP will be provided copies of the applicable SAP sections. Their signatures or email receipt date will indicate that they have read the applicable SAP sections and will perform the tasks as described. If only a portion of the SAP was reviewed, then personnel should note which sections were reviewed.

Name	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Dabra Seiken	Tetra Tech/PM/general project management	978-474-8400	See Worksheet #1	All	
Robin Clark	Tetra Tech/FOL/Project Geologist, and SSO	978-474-8400		All	
Tom Johnston	Tetra Tech/QAM/quality assurance management, data quality review oversight	412-921-8615	See Worksheet #1	All	
Kelly Carper	Tetra Tech/Project Chemist/ laboratory procurement oversight, data quality review, and chemistry support	412-921-7090		All	
Amy Carey	Tetra Tech/Field Sample Collection Specialist/ Sample collection, shipment	978-474-8400		All	
Gary Glennon	Tetra Tech/Database specialist/Geographic Information System (GIS) and analytical data presentation and analysis	978-474-8400		All	
Kelly Perkins	Katahdin/PM/Manages project	207-874-2400		Worksheets #15, 19, 20, 23, 24, 25, 28, 30, 34	
Nilo Ligi	TestAmerica/PM/Manages project	916-374-4427		Worksheets #15, 19, 20, 23, 24, 25, 28, 30, 34	

SAP Worksheet #5 -- Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1)



SAP Worksheet #6 -- Communication Pathways

(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
Regulatory Agency Interface	Tetra Tech PM	Dabra Seiken	978-474-8400	PM will notify the EPA and RIDEM RPMs at least 48 hours prior to commencement of field activities and 24 hours prior to a change in schedule. PM will provide regulators with weekly field updates via e-mail, including activities performed that week and a schedule of planned activities for the following week. PM will notify regulators via e-mail within 48 hours after receipt of a signed concurrence letter from the Navy RPM to change the scope of work, and prior to execution of the work.
Scheduling of utility clearances and access to base	FOL	Robin Clark	978-474-8400	FOL coordinates by phone (or e-mail) with Dig Safe and Naval Station Newport Utility clearance prior to sampling activities
SAP amendments	Navy RPM	Roberto Pagtalunan	757-341-2010	Navy RPM sends scope change within 1 week of recognizing need for SAP amendment to Tetra Tech Program office prior to implementing any changes in scope.
Changes in schedule	Tetra Tech PM	Dabra Seiken	978-474-8400	FOL informs PM by phone within same day of recognizing need for change; PM informs RPM by phone within 24 hours and prepares schedule concurrence letter, if deemed necessary by the RPM and PM.
Issues in the field that result in changes in scope of field work	Tetra Tech FOL	Robin Clark	978-474-8400	FOL informs Tetra Tech PM by phone within same day of identifying field issue. PM approves change same day, if warranted. Document via FMR form, within 2 business days.
Issues in the field that result in changes in scope of work	Tetra Tech FOL Tetra Tech PM	Robin Clark Dabra Seiken	978-474-8400 978-474-8400	FOL informs PM by phone within same day of identifying issue; PM informs RPM by phone within 24 hours, if warranted. PM sends a concurrence letter to Navy RPM, if warranted, within 7 days. RPM signs the letter within 5 days of receipt. Scope change is to be implemented before work is executed. Document the change on a FMR form.

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or e-mail	Procedure
Recommendations to stop work and initiate corrective action	Tetra Tech FOL/SSO Tetra Tech PM Tetra Tech QAM Navy RPM	Robin Clark Dabra Seiken Tom Johnston Roberto Pagtalunan	978-474-8400 978-474-8400 412-921-8615 757-341-2010	Responsible Party informs PM, FOL, and subcontractors verbally within 1 hour on recommendation to stop work and within 24 hours of recommendation to restart work. Responsible party follows verbal notification with an email to Project Team within 24 hours.
Analytical data quality issues	Katahdin PM TestAmerica PM Tetra Tech Project Chemist Navy RPM	Kelly Perkins Nilo Ligi Kelly Carper Roberto Pagtalunan	207-874-2400 916-374-4427 412-921-7090 757-341-2010	The Laboratory PM will notify (verbally or via e-mail) the Tetra Tech Project Chemist within one business day of when an issue related to laboratory data quality is discovered. The Tetra Tech Project Chemist will notify (verbally or via e-mail) the data validation manager (DVM) and the Tetra Tech PM within one business day. Tetra Tech Project Chemist notifies Tetra Tech PM verbally or via e-mail within 48 hours of validation completion that a non-routine and significant laboratory quality deficiency has been detected that could affect this project and/or other projects. The Tetra Tech PM verbally advises the NAVFAC RPM within 24 hours of notification from the project chemist. The NAVFAC RPM takes corrective action that is appropriate for the identified deficiency. Examples of significant laboratory deficiencies include data reported that has a corresponding failed tune or initial calibration verification. Corrective actions may include a consult with the NAVFAC Navy Chemist.

Note: Telephone notifications to be documented via email.

SAP Worksheet #7 -- Personnel Responsibilities Table

(UFP-QAPP Manual Section 2.4.3)

Name	Title/Role	Organizational Affiliation	Responsibilities
Roberto Pagtalunan	RPM/Manager	Navy, NAVFAC Mid Atlantic	Oversees project implementation, including contract management. scoping, data review, and evaluation.
Kymberlee Keckler	EPA RPM/Manager	USEPA Region I	Participates in scoping, data review, evaluation, and review of the SAP. Oversees project execution for USEPA.
Pamela Crump	RIDEM RPM/Manager	RIDEM, Division of Site Remediation	Participates in scoping, data review, evaluation, and review of the SAP. Oversees project execution for RIDEM.
Dabra Seiken	PM/Lead Hydrogeologist	Tetra Tech	As PM, oversees project, financial, schedule, and technical day to day management of the project. Provides technical review of interpreted data. As Lead Hydrogeologist, supervises field work and preparation of geological interpretation and text.
Robin Clark	FOL/Project Geologist/SSO	Tetra Tech	As FOL, supervises, coordinates, and performs field sampling activities. As Project Geologist, assimilates geological data, prepares geological interpretation and text. As SSO, is responsible for staff training and monitoring site conditions related to personnel safety. Details of the SSO's responsibilities are presented in the site-specific Health and Safety Plan (HASP).
Tom Johnston	QAM/Manager	Tetra Tech	Ensures quality aspects of the CLEAN program are implemented, documented, and maintained.
Matt Soltis	Health and Safety Manager (HSM)/Manager	Tetra Tech	Oversees Tetra Tech CLEAN Program Health and Safety Program.
Joe Samchuck	Project Chemist/Technical reviewer	Tetra Tech	Participates in project scoping, prepares laboratory scopes of work, and coordinates laboratory-related functions with laboratory. Oversees data quality reviews and quality assurance of data validation deliverables.
Kelly Perkins	Laboratory PM/Manager	Katahdin	Coordinates analyses with laboratory chemists, ensures that scope of work is followed, provides Quality Assurance (QA) of data packages, and communicates with Tetra Tech project staff.
TBD	Laboratory PM/Manager	Test America	
Gary Glennon	Data Manager	Tetra Tech	Consolidates data in database. Analyzes and presents analytical data. Maps or oversees mapping of data in GIS or other system.
Amy Carey	Staff scientists/Technical support	Tetra Tech	Collect, package, and ship samples in accordance with the SAP. Assimilates analytical data and prepares text regarding nature and extent of contamination.
Nicole Cofrin	GIS Specialist/Graphics support	Tetra Tech	Map data in GIS.

SAP Worksheet #8 -- Special Personnel Training Requirements Table

(UFP-QAPP Manual Section 2.4.4)

Each site worker will be required to have completed appropriate Hazardous Waste Operations and Emergency Response (HAZWOPER) training specified in Occupational Safety and Health Administration (OSHA) 29 Code of Federal Regulations (CFR) 1910.120 (e). Project-specific safety requirements are addressed in greater detail in the site-specific HASP.

SAP Worksheet #9 -- Project Scoping Session Participants Sheet
(UFP-QAPP Manual Section 2.5.1)

Project Name: Data Gaps Assessment Projected Date(s) of Sampling: November to December 2012 _____ Project Manager: Dabra Seiken	Site Name: Tank Farm 3, NAVSTA Newport Site Location: Portsmouth, Rhode Island

Date of Agreement: October 21, 2010
Purpose: Develop project quality objectives using EPA DQO process

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Roberto Pagtalunan	RPM	NAVFAC Mid-Atlantic	757-341-2010	roberto.pagtalunan@navy.mil	RPM
Winoma Johnson	RPM	NAVFAC Mid-Atlantic	757-341-2008	Winoma.johnson@navy.mil	RPM and Team Leader
Stephen Parker	PM	Tetra Tech	978-474-8400	Stephen.Parker@tetratech.com	Facility coordinator

Comments/Decisions: Agreed to approach site using same methodology as Tank Farm 4/5 sites, where hazardous materials releases are addressed as Category 1 under CERCLA, and fuel / petroleum and related releases are addressed as Category 2 under RIDEM Underground Storage Tanks (UST) regulations. It was agreed that petroleum hydrocarbons would not be in the sampling and analysis plan in the Category 1 areas because the components of petroleum hydrocarbons that contribute to the site risk, are quantified by sampling and analysis for the specific components. At the tank farm sites, area on concerns (AOCs) where uncontrolled burning of sludge is suspected will be Category 1 areas.

Use of Category 3 is uncertain.

Action Items: None

Consensus Decisions: None

Project Name: Data Gaps Assessment Projected Date(s) of Sampling: November to December 2012 _____ Project Manager: Dabra Seiken	Site Name: Tank Farm 3, NAVSTA Newport Site Location: Portsmouth, Rhode Island

Date of Session: January 13, 2011
Scoping Session Purpose: Establish concurrence on the need for sampling and analysis at AOC 001 (former burn chamber) and develop framework for sampling and analysis.

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Roberto Pagtalunan	RPM	NAVFAC Mid-Atlantic	757-341-2010	roberto.pagtalunan@navy.mil	RPM
Robert Lim	RPM	USEPA	617-918-1392	lim.robert@epa.gov	RPM
Gary Jablonski	RPM	RIDEM	401-222-2797	Gary.Jablonski@dem.ri.gov	RPM
Dabra Seiken	PM	Tetra Tech	978-474-8445	dabra.seiken@tetratech.com	PM

Comments/Decisions: In EPAs September 29, 2010, comments on the Draft Study Area Screening Evaluation (SASE), the EPA stated that sediment additional characterization is required at AOC 001 to fill a data gap.

- In the Navy's January 13, 2011, response to EPAs comments on the Draft SASE, the Navy agreed to collect additional soil samples to characterize this AOC.
- In the EPAs February 17, 2011, letter, responses to Navy's responses to comments, were provided which indicated that sampling around the former burn chamber along the discharge line from the former burn chamber would be required, and Polynuclear Aromatic Hydrocarbons (PAHs) and dioxins should be included as analytes.
- In the Navy's April 28, 2011, response to the EPAs February 17, 2011 letter, the Navy agreed to sampling and analysis described in the EPAs February 17, 2011, letter.

Action Items: None

Consensus Decisions: None

Project Name: Remedial Investigation (RI) Projected Date(s) of Sampling: November to December 2012 _____ Project Manager: Dabra Seiken	Site Name: Tank Farm 3, NAVSTA Newport Site Location: Portsmouth, Rhode Island

Date of Session: November 17, 2010
Scoping Session Purpose: RPM Meeting. Determine under which regulatory program AOC 20 (transformer area) will be investigated and which media will be targeted for sampling and analysis.

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Dabra Seiken	Project Manager	Tetra Tech	978-474-8400	Dabra.Seiken@tetratech.com	PM, Lead Geologist
Roberto Pagtalunan	RPM	NAVFAC Mid-Atlantic	757-341-2010	roberto.pagtalunan@navy.mil	RPM
Kymberlee Keckler	RPM	USEPA	617-918-1385	kymberlee.keckler@epa.gov	RPM
Stephen Parker	PM	Tetra Tech	978-474-8400	Stephen.Parker@tetratech.com	Facility coordinator
Gary Jablonski	RPM	RIDEM	401-222-2797	Gary.Jablonski@dem.ri.gov	RPM

Comments/Decisions:

- Navy provided a recommendation in the July 2010 Draft SASE, regarding which areas required further investigation under CERCLA-regulations (Category 1) and under the RIDEM Division of Site Remediation (Category 3).
- In the November 17, 2010, RPM meeting the EPA stated that they believed the investigation of the transformer area should be performed under CERCLA authority (Category 1).
- In the Navy's January 13, 2011, response to EPAs comments on the Draft SASE, the Navy agreed to perform the investigation of the transformer area under CERCLA authority (Category 1), and agreed to collect additional soil sampling for polychlorinated biphenyls (PCBs) for characterization of this area.
- In the EPA's February 17, 2011, responses to the Navy's January 13, 2011, Response to Comments (RTCs), the EPA indicated that groundwater sampling for PCBs would be warranted in the transformer area.
- In the Navy's April 28, 2011, response to the EPAs February 17, 2011 letter, the Navy agreed to groundwater PCB sampling and analysis in the transformer area.

Action Items: None

Consensus Decisions: None

Project Name: RI Projected Date(s) of Sampling: November to December 2012 _____ Project Manager: Dabra Seiken	Site Name: Tank Farm 3, NAVSTA Newport Site Location: Portsmouth, Rhode Island

Date of Session: March 16, 2011
Scoping Session Purpose: Establish concurrence that investigation is warranted in the Building 227 Electical Control House (ECH) area

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Dabra Seiken	PM	Tetra Tech	978-474-8400	Dabra.Seiken@tetratech.com	PM, Lead Geologist
Roberto Pagtalunan	RPM	NAVFAC Mid-Atlantic	757-341-2010	roberto.pagtalunan@navy.mil	RPM
Kymberlee Keckler	RPM	USEPA	617-918-1385	kymberlee.keckler@epa.gov	RPM
Gary Jablonski	RPM	RIDEM	401-222-2797	Gary.Jablonski@dem.ri.gov	RPM

Comments/Decisions:

- In an email from RIDEM (dated December 23, 2010), RIDEM indicated that Building 227 should be investigated for releases to the environment.
- In an email response (dated March 3, 2011) from Tetra Tech, Navy agreed to investigate this area by performing limited sampling and analysis of soil in the vicinity of Building 227.
- During the March 16, 2011, RPM meeting, a general concurrence was reached that the investigation of Building 227 would be performed under CERCLA authority because it is a former ECH and the potential contaminants would be CERCLA regulated.
- In an email from RIDEM (dated March 23, 2011) RIDEM indicated that Building 227 plans should be reviewed, the inside and outside of the building should be investigated for possible sources and sampling and analysis should include PCBs, mercury, arsenic, and lead at any and all entrances and on all four sides of the building because these chemicals are potentially related to site operations.

Action Items: None

Consensus Decisions: None

Project Name: RI Projected Date(s) of Sampling: November to December 2012 _____ Project Manager: Dabra Seiken	Site Name: Tank Farm 3, NAVSTA Newport Site Location: Portsmouth, Rhode Island

Date of Agreement: January 13, 2011
Purpose: Establish concurrence that further investigation is warranted in Lawton Brook.

Name	Title	Affiliation	Phone #	E-mail Address	Project Role
Dabra Seiken	PM	Tetra Tech	978-474-8400	Dabra.Seiken@tetratech.com	PM, Lead Geologist
Roberto Pagtalunan	RPM	NAVFAC Mid-Atlantic	757-341-2010	roberto.pagtalunan@navy.mil	RPM
Robert Lim	RPM	USEPA	617-918-1392	lim.robert@epa.gov	RPM
Gary Jablonski	RPM	RIDEM	401-222-2797	Gary.Jablonski@dem.ri.gov	RPM

Comments/Decisions:

- In EPA's September 29, 2010, comments on the Draft SASE, the EPA stated that sediment sampling would be necessary in Lawton Brook to fill a data gap.
- In the Navy's January 13, 2011, response to EPAs comments on the Draft SASE, the Navy agreed to collect limited samples of sediment at Lawton Brook.
- In the EPAs February 17, 2011, letter, responses to Navy's responses to comments, were provided which indicates that sediment downstream of the culvert under Defense Highway should be sampled.
- In the Navy's April 28, 2011, response to the EPAs February 17, 2011 letter, the Navy indicated a sediment sample would be located downstream of the outfall from AOC 001.

Action Items: None

Consensus Decisions: None

SAP Worksheet #10 -- Conceptual Site Model
(UFP-QAPP Manual Section 2.5.2)

10.1 SITE LOCATION AND BACKGROUND

Tank Farm 3 ("the Site") is located on the western shore of Aquidneck Island, in the southwestern portion of Portsmouth, Rhode Island, just north of Newport, Rhode Island (Figure 1). The site topography slopes from a high of approximately 100 feet above mean low water (MLW) elevation in the south central portion of the Site, to a low of approximately 40 feet above MLW on the northwest side of the Site (along Defense Highway), and to a low of approximately 10 feet above MLW on the northeastern side of the Site (along Lawton Brook). Lawton Brook flows northwesterly across the Site, flows under Defense Highway in culverts, and discharges into Narragansett Bay, about 300 feet to the west. Groundwater flow at the Site is to the north and northeast.

The Site (delineated by the property boundary in Figure 2) encompasses approximately 40 acres and is bordered by Defense Highway to the northwest, Raytheon's Submarine Signal Division plant to the northeast, Bayview Estates (residential condominiums) to the southeast and newer residential properties to the southwest.

The site consists of an upland area in the south central portion of the site and a wetland area located along the eastern/northeastern boundary. A wooded area exists between the wetlands and Lawton Brook and the upland portion of the site where the tanks are situated. There are miscellaneous structures at the Site and the ground surface is covered in vegetation, such as brush and grasses, with a few clear areas along paved access roads. Access to the property is via Defense Highway, which runs along the western border, between the property and Narragansett Bay.

The Site has five 1.18 million gallon-capacity, concrete, USTs and two 2.1 million gallon-capacity steel USTs. These tanks formerly were used to store aviation fuels (jet propulsion [JP]-4, JP-5, and JP-8) and marine diesel fuel. The Site also contains appurtenances and support buildings/structures associated with petroleum storage and distribution (underground fuel distribution lines installed roughly 4-feet below grade; an underdrain system (ring drain) around each UST that collects and transfers excess groundwater away from the USTs; sump pump chambers in the pump house next to each UST that contain pumps for the fuel distribution lines and the ring drain; buried piping connecting the fuel distribution lines and the pump houses; and a UST vent and a gauging house connected to each UST.

In their investigation of Tank Farm 3 the DESC, working with RIDEM, reviewed historic aerial photography to identify other areas at each tank farm for investigation/remediation; these are identified as AOC 001 through AOC 033. Additionally, RIDEM provided other areas of concern to be investigated/remediated. Appendix A provides a summary of the identified AOCs at Tank Farm 3.

In accordance with decisions made by the project team (Worksheet #9), the AOCs at the Site have been separated into Category 1 (CERCLA- regulated) areas, Category 2 (RIDEM UST Division regulated) areas, and Category 3 areas (regulatory pathway not yet defined). This SAP has been prepared to address the Category 1 areas only.

The categories of areas within the tank farms are defined as follows:

1. Category 1 (CERCLA-regulated) areas – these are locations of releases/suspected releases of CERCLA hazardous substance(s), which were not the result of DESC's petroleum operations.

2. Category 2 (RIDEM UST Division regulated) areas – these locations relate to petroleum contamination resulting from DESC operations, and which have been, or are currently being, addressed by DESC.

Category 3 areas (additional areas of concern) – areas for which the scope of investigation has not been determined, a release is not confirmed, and a regulatory pathway is not yet defined.

The Category 1 areas consist of two types of areas/contaminants at Tank Farm 3:

1. Contaminants associated with the burning of tank sludge (PAHs, metals, and dioxins). Water containing organic sludge was filtered through a sand filter in the burn pit. Sludge from storage tank bottoms was sometimes burned in this pit (and sometimes removed and disposed elsewhere) when it had accumulated sufficiently to clog the filter. Burning sludge can alter the petroleum sludge, potentially producing dioxins and pyrogenic PAHs, and release elevated concentrations of heavy metals, all of which may have deposited onto soil. Heavier petrogenic PAHs that are not combustion related products may also have been released by this process to surface soil. The USEPA has stated that areas where evidence of sludge burning has taken place would be governed by CERCLA.

2. Areas where PCBs or metals may have been released to the environment. The USEPA has stated that the areas where PCB- containing oils were used would be governed by CERCLA.

The Category 1 portions of the Site that have not been adequately characterized with respect to PAHs, metals, dioxins and/or PCBs and require further investigation are:

1. Building 227 (ECH)

2. AOC 001 (Former Sand Filter/Burning Chamber, its discharge line, and discharge location [wetland])

3. AOC 020 (Electrical Transformer Area)

10.2 SITE HISTORY

The US Navy has owned the Site since at least the 1940s. USTs were constructed in the 1940s and stored virgin petroleum from the early 1940s until 1998. The tanks still remain on Site although they have not been used for fuel storage since tank closure activities were performed between 1996 and 2000. The Site was operated by the Navy until 1974, when the property was leased to the DESC. The DESC actively operated the Site until the 1990s, when the tanks were emptied and cleaned. The DESC still maintains contractual control of the Site, although it is not in active operation.

Building 227 (ECH)

This structure houses electrical equipment, including a transformer, for the operation of the tank farm. This structure is shown on the 1954 Fuel Distribution Plan, suggesting the structure is more than 56 years old. It is presumed that batteries of an unknown nature have been stored in the building. The electrical equipment in this structure has reportedly been out of service for more than 15 years. Based on the former use of Structure 227, the potential contaminants in this area are PCBs that may have been present in the transformer oil, and metals that may have been present in the batteries.

AOC 001 (Former Sand Filter/Burn Chamber)

The USTs at the Site were periodically cleaned and the bottoms of the tanks were pumped to remove accumulated sediments and water (sludge). Prior to 1974, tank bottom sludges were pumped to the sand filter. Residual petroleum sludge remaining in the sand filter was either burned in the sand filter structure, or scraped off and removed to an off-site location. The filtered water appears to have discharged into the Lawton Brook/wetland area at the site that ultimately discharges into Narragansett Bay. Prior to the installation of the oil-water separator (OWS) (presumed to be circa 1974), the sand filter was also routinely used for the discharge of groundwater from the ring drains associated with the USTs. Based on the former use of the sand filter as a burning chamber and a discharge area for groundwater around the USTs, the potential contaminants in this area are volatile organic compounds (VOCs), PAHs, metals, and dioxins.

Also located within the boundaries of AOC 001 is the location of a former “stripper valve point”. This structure was part of the petroleum distribution piping that was previously cleaned and decommissioned. It was sometimes called a “stripper pit” because of the presence of a pit that contained centrifugal stripper pumps that removed any water from the fuel via centrifugal force. There is no record of a release at this location and it has been previously closed out. This area will be observed and investigated during the investigation of AOC 001.

AOC 020

AOC 20 consists of the location of a former transformer blockhouse that was replaced in 1980 with two new transformers. Both of these transformers are mounted on concrete pads and have been out of

service for more than 15 years. Based on the former use of this area for electrical equipment and the presence of transformers, the contaminants in this area are PCBs that may have been present in transformer oil.

10.3 GEOLOGY

The Site is located in the southeastern portion of the Narragansett Basin which is underlain by Pennsylvanian age, non-marine, sedimentary and metamorphic rocks, including the Rhode Island Formation. The overburden site geology is generally characterized (GZA, 1995; GZA, 1996) as glacial till between 4 and 25 feet thick and which consists of a medium to very dense unsorted mixture of sand, gravel, and silt with occasional layers of silty sand and clayey sand or boulders. Loose glacial till was encountered in some areas and is likely re-worked till that was used as fill. In the central portion of the site a loose, silty fine sand strata was encountered from the ground surface to between 3 to 5 feet, and the depth to bedrock was noted as shallow (from 4 to 11 feet).

Bedrock at the Site is described mostly as metamorphosed shale with occasional strata of siltstone, sandstone, conglomerate, schist, phyllite, slate, and quartz. The upper portion of the bedrock (3 to 20 feet) is highly weathered and highly fractured in some locationswith fracturing generally decreasing with depth.

The water table elevation is usually encountered in the bedrock or at the bedrock/overburden interface. Groundwater flows northerly in the western portion of the site. Groundwater flows easterly or northeasterly in the eastern portion of the site, towards Lawton Brook.

10.4 SUMMARY OF ENVIRONMENTAL WORK CONDUCTED

The first environmental investigation of the Site and other areas was an Initial Assessment Study (IAS), completed by Envirodyne Engineers Inc. in 1983. On completion of the IAS, the Site was recognized as an area that required further environmental investigation, because petroleum tank bottom sludge was burned in a burning chamber (the sand filter) and this process could have resulted in release of contaminants to the environment. No samples for laboratory analysis and reporting were collected from the Site as part of the IAS.

Various reports on investigations, remediation, and closures were generated to document activities undertaken between 1992 and 2001. These activities primarily had to do with the storage and distribution of virgin petroleum products; however, some of the work conducted relates to the three Category 1 areas of the Site currently planned for investigation. These reports include:

- Progress Report on Initial Assessment Conducted in the Vicinity of Tank 70 (GTI, 1992)
- Environmental Site Investigation (GZA, 1995)
- Supplemental Site Investigation (GZA, 1996)
- Supplemental Site Investigation and Corrective Action Plan (GZA, 1998a)

- Draft Tank Closure Assessment and Report (September, 1998b)
- Draft Underground Fuel Line Closure and Soil Gas Survey Report (GZA, 1998c)
- Tank Farm 3 Soil Removal Activities in the Vicinity of Tank 35 (FwEC, 2000)
- Closure Report for Underground Storage Tanks at Tank Farm 3 (FwEC, 2001)
- Site Investigation Remedial Action Report (TtEC, 2005)

AOCs were identified at the Tank Farm 3 site using aerial photography during the Site Investigation and Remedial Action Report (SIRAR) activities completed between 2004 through 2005 (TtEC, 2005). Once these AOCs were field verified, soil samples were collected and analyzed for total petroleum hydrocarbon (TPH) in the field using Petroflag field analytical methodologies. If Petroflag results were identified at concentrations greater than 100 parts per million (ppm), the samples were analyzed in a laboratory for TPH using EPA Method 8015 for diesel range and gasoline range organics (DRO and GRO). If Petroflag or laboratory analysis indicated the TPH concentration was less than 100 ppm, additional analyses or remediation efforts were not performed. If laboratory results indicated the TPH concentration was between 100 and 500 ppm, the corresponding samples were analyzed in a laboratory for VOCs and semi-volatile organic compounds (SVOCs) for comparison to RIDEM's Direct Exposure Criteria (DEC). The soil cleanup goal was set as RIDEM's industrial DEC. Soils with analyte concentrations greater than the cleanup goal were excavated and disposed of off-site.

In the course of reviewing the various investigations described above, three areas requiring further testing/exploration under CERCLA were identified. A description of the investigations previously performed in these areas and the associated findings are provided below.

10.4.1 Former Sand Filter/Burn Chamber (AOC 001)

Two soil borings were advanced at Tank Farm 3 near the Former Concrete Sand Filter structure (Figure 3). GZ-301, hydraulically down-gradient of the structure, was advanced during the Environmental Site Investigation. GZ-332, hydraulically upgradient of the structure, was advanced during the Supplemental Site Investigation and Corrective Action. Soil samples were collected and soil sample headspace was screened for total volatile organics (TVOCs) at 5 foot intervals. The sample collected from 0.2 to 2.2 feet below ground surface (bgs) at GZ-301 was analyzed for TPH, total volatile petroleum hydrocarbons (TVPH) and VOCs. TVPH and VOCs were not detected. The results of the TPH analysis indicated the presence of a low concentration (28 ppm) of TPH in the sample. The sample collected from 5 to 6 feet bgs at GZ-332 was analyzed for TPH, VOCs, and PAHs, and none of these constituents were detected.

The soil borings mentioned above were completed as groundwater monitoring wells. Groundwater from GZ-301 was collected on three occasions. The sample was analyzed for TPH, TVPH, and VOCs during the Environmental Site Investigation; the sample was analyzed for PAHs, VOCs, TPH, and TVPH during the Supplemental Site Investigation, and during the SIRAR, the sample was analyzed for VOCs, SVOCs,

and DRO. Groundwater from GZ-332 was sampled on two occasions. During the Supplemental Site Investigation and Corrective Action, the sample was analyzed for VOCs, TVPH, and TPH and during the SIRAR, the sample was analyzed for VOCs, SVOCs, and DRO. GRO was the only constituent detected in the groundwater samples. GRO was detected between 0.272 and 1.3 milligram per liter (mg/L) in GZ-301. GRO was detected at 0.14 mg/L in GZ-332.

During the SIRAR, the sand filter pit was located under a road. The outlet pipe for the sand filter pit appears to have discharged into the Lawton Brook wetlands at the Site (Figure 3). Road asphalt and up to 10 feet of fill material beneath the road was excavated as part of efforts to expose the sand filter pit. The fill (concrete rubble, wood, cobbles, sand, and silt) was removed and the sand filter pit was exposed. The visibly contaminated oily material from inside the sand filter pit was excavated and stockpiled using an excavator and a vacuum truck. The walls and floor of the sand filter pit were pressure washed and the rinsate water was removed for disposal. Test pits were excavated adjacent to each side of the sand filter pit, and soil samples were collected from 2 feet below the top of the pit walls and from below the base of the pit wall. Soil samples were also collected beneath pipes associated with the sand filter pit. Six soil samples were sent to the laboratory for analysis of DRO and GRO. Of the six samples, five had detections of GRO, while none had detections of DRO. In TF3-001-S1-2.0, collected on the west side of the structure, GRO was present at 6,582 milligram per kilogram (mg/kg) and was collected at a seam in the concrete wall. However, a sample (TF3-001S1-2.0A) was collected 2 feet west of TF3-001-S1-2.0 and only indicated a GRO concentration of 13 mg/kg. Sample TF3-001-S3-2.5 indicated a GRO concentration of 4,133 mg/Kg. This sample was collected below the intake pipe, on the south side of the structure. Soil samples collected on the north and east sides of the structure contained only low concentrations (26 to 45 mg/kg) of GRO.

Three surface water samples (SW-1, SW-2, and SW-3) were collected from Lawton Brook during implementation of the Environmental Site Investigation at the Site. The surface water samples were analyzed for TPH, TVPH, and VOCs. SW-1 was located as an upstream sample that would have not been impacted by site operations. SW-2 was located as a mid-stream sample, approximately 1,000 feet upstream of the culvert that runs beneath Defense Highway. SW-3 was located as the downstream sample, immediately upstream from the culvert that runs beneath Defense Highway (Figure 2). The surface water results indicated chlorinated VOCs to be present in all three samples, and TVPH in two of the three samples. These contaminants were not attributed to the Site at that time, but rather were attributed to an un-named off-site source. Of note is the observation that the site is bounded to the northeast by Raytheon's Submarine Signal Division and the ground surface from this adjoining property slopes southwest towards Lawton Brook.

Although unrelated to the former sand filter/burn chamber, an emergency response was performed in 2008 within AOC 001 to address a release of petroleum. In April of 2008 a boater observed sheen on Narragansett Bay in the vicinity of Lawton Brook. This incident was reported to RIDEM and the U.S. Coast Guard who subsequently contacted the U.S. Navy. The Navy authorized an emergency response

company to place booms along the brook and in Narragansett Bay to contain the oil/sheen. The Navy then initiated excavation to investigate the sheen. It was discovered that a 12-inch pipe located along Defense Highway had leaked free petroleum product (assumed to be jet fuel) into the pipe chase and subsequently into Lawton Brook.

A removal action was initiated to remediate the contaminated soil from this release and included the following tasks:

- removal of the overlying soil, and testing of that soil for use as backfill.
- Removal of pipes and the pipe chase.
- Asbestos abatement of insulation surrounding the pipeline.
- Capping/plugging of the remaining ends of the removed pipes.
- Excavation of contaminated soils.
- Excavation backfill.
- Site restoration.

During the excavation of contaminated soil, confirmatory Petroflag™ soil samples were collected every 20 linear feet along sidewalls, and every 10 feet along the excavation bottom. Excavation continued until observation and field screening results indicated that clean material had been reached.

10.4.2 Electrical Control House (Building 227)

One soil boring (GZA-328) was advanced at Tank Farm 3 at the ECH during the Supplemental Site Investigation at a location illustrated in Figure 4. Soil samples collected from this soil boring advancement were screened at 5 foot intervals. The sample collected from 0 to 2 feet bgs was selected for laboratory analysis based on soil headspace screening for TVOCs that either indicated the potential presence of organic contaminants or because it was selected as a default sample when no elevated headspace measurement was observed. The sample was analyzed for TPH, VOCs, and PAHs, with all laboratory results identified as non-detected above the laboratory reporting limit.

GZ-328 was constructed as a groundwater monitoring well and sampled on three occasions. The first sampling event at this monitoring well occurred during the Supplemental Site Investigation (GZA, 1996) and was submitted for laboratory analysis and reporting for PAHs, VOCs, TPH, and TVPH. The second sampling event was undertaken during a groundwater sampling event in June 1999 when the sample was submitted for TPH, VOCs, and SVOCs analysis. The third sampling event at this monitoring well occurred during the SIRAR (TtEC, 2004) when the sample was submitted for VOCs, SVOCs, and DRO analysis. Groundwater analytical results from these three sampling events did not identify any constituents above the laboratory reporting limits.

A potential pipe leading from the south side of the ECH was identified from examination of a 1988 aerial photograph. This potential pipe was investigated as part of the SIRAR (TtEC, 2005) and included test

pitting, a magnetometer survey in the test pits, and soil screening in the area in which the potential pipe was identified. Soil samples were screened for TPH using Petroflag™ methodology. These screening results indicated TPH values less than 100 ppm and, as such, this area was not evaluated further.

10.4.3 Transformer Area (AOC 020)

One soil boring (GZA-314) was advanced in the area of Outdoor Electrical Transformers during the Environmental Site Investigation at Tank Farm 3 at a location depicted on Figure 5. Samples were collected and soil headspace was screened for TVOCs at 5 foot intervals from soil boring GZA-314. Based on headspace screening results indicating the potential presence of organic contaminants or because it was selected as a default sample when no elevated headspace measurement was observed, the sample collected from the 0 to 2 foot interval was submitted for TPH, TVPH, VOCs, PCBs, and pesticides laboratory analysis and reporting. Laboratory results from this sample did not identify any constituents above the laboratory reporting limit.

Soil boring GZ-314 was subsequently completed as a groundwater monitoring well and sampled on at least four occasions. The first sampling event was undertaken as part of the Environmental Site Investigation where the collected sample was analyzed for TPH, TVPH, VOCs, and PCBs; laboratory results indicated that these constituents were not detected. The monitoring well was again sampled during the Supplemental Site Investigation where the collected sample was analyzed for PAHs; laboratory results from this effort indicated that these constituents were not detected. As part of the groundwater sampling event in June 1999, the sample was analyzed for TPH, VOCs, and SVOCs; again, laboratory results indicated that these constituents were not detected. As part of the field work for the SIRAR, the sample was analyzed for VOCs, SVOCs, and DRO; laboratory results again indicated that these constituents were not detected.

An aerial photograph dated 1986 was reviewed during the SIRAR, which suggested that there was a probable concrete structure located in this area. This area was subsequently designated as AOC 020. To investigate AOC 020, test pitting, magnetometer surveying of the test pits, and soil sampling were performed (TtEC, 2005). Soil sampling was performed using criteria described in Section 10.4. VOCs, SVOCs, and TPH concentrations did not exceed screening criteria in the four soil samples sent to the laboratory as part of test pitting.

Additional sampling and analysis was performed around each of the two pad mounted transformers, as part of the SIRAR (TtEC, 2005). Four samples were collected around transformer #1 (TF3-TF1-A, B, C and D) and four samples were collected around transformer #2 (TF3-TF2-A, B, C and D) at locations depicted in Figure 5. The eight soil samples from around the two pad mounted transformers in this AOC were analyzed for PCBs. Of the eight soil samples from around the transformers in this AOC, three surface samples (TF3-TF1-D, TF3-TF2-A, and TF3-TF2-C) contained concentrations of Aroclors 1242, 1254, and 1260 greater than reporting limits. Each of the total PCB concentrations (1210, 1450, and

8200 microgram per kilogram [µg/kg]) also exceeded the Industrial ORNL RSL of 740 µg/kg for total PCBs.

10.5 CONCEPTUAL SITE MODEL

Figure 6 presents the conceptual site model (CSM) for the Site. The CSM is also described below:

- Potential contaminants associated with the burning of petroleum sludge (PAHs, dioxins and furans [hereafter referred to as "dioxins"], and metals) were likely released by burning sludge in the sand filter/burn pit (AOC 001). Although fuels formerly stored in the Tanks contain only trace amounts of heavy metals, these trace concentrations become concentrated in tank bottom sludges and become even more concentrated following combustion of the sludge. Furthermore, the sand filter/burn pit was also routinely used to collect discharge of the groundwater from the ring drains (prior to the installation of the OWS). Potential contaminants associated with this use of the sand filter include petroleum-related VOCs and PAHs.
    - Prior to its cleaning, constituents could have migrated, dissolved in water, from the sand filter/burn pit and associated discharge line, through cracks in the structures and could have contaminated soil surrounding the concrete structure and the discharge line.
    - Constituents could dissolve from the sludge and migrate vertically downward, potentially reaching groundwater. These contaminants could migrate in groundwater downgradient to the northeast, which is the direction of groundwater flow. Groundwater testing in monitoring well GZ-301, located about 25 feet downgradient northeast of this area, indicates that groundwater has not been impacted by sludge burning. However, groundwater is to be re-tested to confirm these results, and to test for additional constituents (metals, PAHs, and VOCs).
    - Constituents could dissolve in water and discharge via the discharge line(s) from the sand filter/burn pit and migrate to the surface water discharge location(s). Previous surface water sampling was conducted and the results did not indicate surface water impacts from the site.
    - Constituents could have been deposited in Lawton Brook sediment from previous surface water discharges from the sand filter/ burn pit.
    - Constituents could migrate in the smoke from the combustion of the sludge. These constituents would have dispersed and been deposited via aerial deposition. The concentrations from this deposition mechanism would be diluted due to the large area over which the deposition would occur and the vast majority of the constituents are expected to be immediately around the former burn chamber.
    - Constituents could also be transported over the ground surface via overland flow following a precipitation event. However, the vast majority of the constituents are expected to be confined to the former burn areas and the area immediately surrounding and beneath the former burn areas.

- Volatile constituents could dissolve in groundwater and volatilize from groundwater to the unsaturated zone and migrate in vapor from the unsaturated zone to the air. Previous groundwater sampling was conducted and did not indicate concentrations of volatile constituents at levels that would be expected to result in detectable concentrations in air from this migration pathway.

- PCBs associated with the electrical transformers at AOC 020 are present in surface soil around the pad mounted transformers. PCBs were likely released from normal use and maintenance activities.
  - PCB- containing oil could have been released to the ground surface (surface soil) and could migrate vertically downward, potentially reaching subsurface soil and groundwater. Although not readily soluble in water, low levels of these PCBs could migrate in groundwater, especially if adsorbed to colloidal matter.

- Potential contaminants (PCBs and metals) associated with the ECH (Building 227) could have been released from the normal use of the building and maintenance of the electrical equipment inside the building.
  - PCB- containing oil or metals from electrical equipment and/or metals from batteries could have been released to the surface and subsurface soil from potential release(s) inside the building and the constituents being transported outside the building via a floor drain. Furthermore, the potential exists for additional building-related sources. Evidence of one subsurface pipe was identified in a 1988 aerial photograph and was investigated during the SIRAR. Soil sampling associated with that potential pipe was performed for TPH only, thus leaving a data gap with regard to other potentially site-related contaminants.

Currently, the site is largely unused and exposure to human receptors is limited. There is some limited hunting allowed by Navy personnel. The current receptors to environmental contamination include terrestrial and aquatic biota and human receptors that could be exposed to impacted surface soil and sediment. Potential human receptors include trespassers and limited recreational users (hunters). Subsurface soil is not currently accessible, as the site is unused and vacant. With the exception of where it discharges into Lawton Brook, groundwater is not currently accessible, as there are no water supply wells at the site. Potential terrestrial ecological receptors, such as plants, soil invertebrates, mammals, birds, and reptiles, can be exposed to contaminated surface soil. While most terrestrial receptors are not substantially exposed to subsurface soils (soil below 12 inches deep), shallow subsurface soil (the top 12 inches) is accessible to some terrestrial receptors. Aquatic ecological receptors, such as fish, sediment invertebrates, reptiles, and amphibians, can be exposed to sediment contamination through direct contact and incidental sediment ingestion. Terrestrial wildlife may also be exposed to the sediment, although to a lesser degree, through direct contact and incidental sediment ingestion. Terrestrial vertebrates may be exposed to contaminated sediment through ingestion of aquatic prey.

Potential future exposure pathways would be exposure to subsurface soil and groundwater to humans via a change in site use, such as site development. Exposure to subsurface soil would be from construction workers' activities involving excavation, including contaminated soil being brought to the surface. Exposure of potential future residents to groundwater would be from the potential installation and use of a groundwater supply well at the Site for drinking water and/or irrigation purposes.

SAP Worksheet #11 -- Project Quality Objectives/Systematic Planning Process Statements
(UFP-QAPP Manual Section 2.6.1)

The following text describes the development of project quality objectives (PQOs) using the USEPA data quality objective (DQO) process. The primary data users of this investigation will be Tetra Tech and the Navy.

11.1 PROBLEM STATEMENTS

AOC 001 – Former Sand Filter/Burn Chamber, piping and wetland – AOC 001 has been identified as having a history of burning of tank bottom sludge inside the concrete sand filter/burn chamber. Burning of petroleum sludge can potentially alter the sludge, releasing dioxins, metals, and PAHs to soils and sediment at concentrations exceeding risk screening criteria. Furthermore, the use of this structure for the discharge of groundwater from the ring drains around the USTs potentially released petroleum-related VOCs and PAHs to soil and sediment via the sand filters discharge line at concentrations exceeding risk screening criteria. These petroleum-related VOCs and PAHs are expected to be commingled with the CERCLA contaminants released during sludge burning. Releases could have resulted in impacts to groundwater beneath and/or hydraulically down-gradient of the structure. The nature and extent of such contamination has not been established. The Project Team determined that contaminants related to AOC 001 will be classified as Category 1, and an investigation and evaluation of environmental media under Category 1 shall be conducted so that, if necessary, a CERCLA risk assessment can be performed. Therefore, data must be collected in accordance with the Navy and USEPA policies for conducting risk assessments under CERCLA. Additionally, regardless of the category of the AOC, RIDEM has requested that TPH data be collected at AOC 001.

In order to determine whether a risk assessment is necessary, the following problems must be resolved:

Problem 1: The Navy must determine the nature and extent of contamination related to burning of sludge and the processing of water from ring drains in soil and sediment at AOC 001, and must estimate whether risks from exposure of human and ecological receptors to site contaminants could be unacceptable so that necessary actions can be taken to further investigate or mitigate the risks, in an effort to protect human health and the environment.

Problem 2: The Navy must determine whether groundwater has been contaminated as a result of sludge burning at AOC 001 so appropriate actions can be taken to mitigate or further investigate the possibility of unacceptable risks to human receptors in an effort to protect human health and the environment.

AOC 020 – Transformer Area – Previous surface soil sampling around the transformers indicated the presence of PCBs at concentrations up to 8200 µg/Kg total PCBs, greater than screening criteria. However, the nature and extent of PCB-contaminated soil must be determined. One groundwater sample available down-gradient of the PCB-contaminated soil has not indicated PCBs in groundwater, however,

this absence of PCB contamination in the groundwater must be investigated further to confirm or refute this earlier finding. New data are needed to represent current conditions in groundwater from the existing well, and in soil at additional locations. The Project Team determined that contaminants related to AOC 020 will be classified as Category 1, and an investigation and evaluation of environmental media under Category 1 shall be conducted so that, if necessary, a CERCLA risk assessment can be performed. Therefore, data must be collected in accordance with the Navy and USEPA policies for conducting risk assessments under CERCLA. In order to determine whether a risk assessment is necessary, the following problems must be resolved:

Problem 1: The Navy must determine the nature and extent of PCB contamination in soil in the vicinity of AOC 020/pad-mounted transformers, and must estimate whether risks from exposure of human and ecological receptors to site contaminants could be unacceptable so that necessary actions can be taken to mitigate such risks in an effort to protect human health and the environment.

Problem 2: The Navy must determine whether groundwater down-gradient of the transformers has been contaminated by the known presence of PCBs in the surface soil so appropriate actions can be taken to mitigate or further investigate the possibility of unacceptable risks to human receptors in an effort to protect human health and the environment.

Building 227 – Electrical Control House – Previous soil sampling around a presumed pipe leading from the ECH was performed for TPH analysis only. TPH was not detected at concentrations greater than 100 ppm utilizing field screening methods. One groundwater monitoring well down-gradient of the ECH did not indicate the presence of VOCs, SVOCs, PAHs, or TPH in groundwater. However, additional investigation is necessary for PCBs and metals to determine if media are impacted by these site-related contaminants. In addition, RIDEM has requested that TPH data be collected at the ECH, and the Navy has agreed to conduct this additional analysis. The Project Team determined that contaminants related to Building 227 will be classified as Category 1, and an investigation and evaluation of environmental media under Category 1 shall be conducted so that, if necessary, a CERCLA risk assessment can be performed. Therefore, data must be collected in accordance with the Navy and USEPA policies for conducting risk assessments under CERCLA. In order to determine whether a risk assessment is necessary, the following problem must be resolved:

Problem: The Navy must determine if PCBs and metal contamination of soil and groundwater is present in the vicinity of the ECH. The Navy must evaluate these site conditions to determine an appropriate course of action to be protective of human health and the environment.

11.2 IDENTIFY INPUTS TO PROBLEM RESOLUTION

The inputs needed to resolve the problems identified in Section 11.1 include field measurements, laboratory chemical data, and Project Screening Levels (PSLs), as described below. Field tasks to be performed to collect these data inputs are summarized in Worksheet #14.

11.2.1 Field Measurements

The following field measurements are needed:

- During monitoring well development, measurement of water quality parameters (temperature, pH, specific conductance, and turbidity) and recording of development details (start and stop times, quantity of water removed, water level changes) must be performed. These data are needed to determine that the well screen is hydraulically connected to the groundwater within the screened interval, indicating that development is complete for each well.
- During groundwater sampling, measurement of water quality parameters must be performed (temperature, dissolved oxygen [DO], pH, specific conductance, oxidation-reduction potential [ORP], and turbidity) from purge water collected from the same wells where groundwater samples will be collected for laboratory analysis. These data are needed to determine that the water brought to the surface is stabilized and representative of the groundwater within the screened interval, indicating that sample collection is appropriate. The final measurements of these parameters made at each well are needed to characterize water quality of that groundwater.
- Water level measurements, such as existing elevation and location data for the existing wells to be sampled; and a survey of the elevations and the locations of newly-installed wells must be collected so that water table measurements can be used to assess groundwater elevations and flow directions. Coordinates will be determined by standard surveying techniques. The vertical datum will be MLW elevation or National Geodetic Vertical Data (NGVD) 1929 depending on the most accessible benchmark. The horizontal coordinates shall be referenced to the State of Rhode Island Grid Coordinate System, North American Datum (NAD 1983).
- Field screening (jar headspace) measurements using a photoionization detector (PID) must be performed to assist in selection of subsurface soil intervals that will be sent to the laboratory for analysis at AOC 001.

11.2.2 Laboratory Chemical Data

The following Category 1 chemical data from fixed-base laboratory analyses are needed and the list of target analytes is presented in Worksheets #15a (soil), #15b (groundwater), and #15c (sediment):

- For AOC 001, concentrations of petroleum-related VOCs, PAHs, TPH, dioxins, and metals in soil (surface and subsurface soil) and sediment are needed, while concentrations of PAHs and metals are needed for groundwater. In addition, data for non-chlorinated VOCs and PAHs are also needed in surface and subsurface soil, sediment, and groundwater because they are components of aviation fuels and/or they are combustion products of the fuels and serve as convenient indicators of petroleum hydrocarbon contamination, which is not regulated under CERCLA. The VOCs and PAHs also represent the more toxic components of petroleum fuels. These analytical groups were identified

as the most likely classes of contaminants associated with the burning of petroleum sludge, and the co-mingling of CERCLA contaminants with petroleum contaminants. These data (except for TPH) are needed to determine if a risk assessment is necessary. TPH data are needed to satisfy the RIDEM. For AOC 020, concentrations of PCBs in surface and subsurface soil and groundwater are needed. This analytical group was identified by the Project Team as the most likely class of contaminants associated with the transformers.

- For Building 227 (the ECH), concentrations of PCBs, TPH and metals in surface and subsurface soil and groundwater (except TPH) are needed. This analytical group was identified by the Project Team as the most likely classes of contaminants associated with the transformer and other uses of the building.

11.2.3 Project Screening Levels

The newly-collected chemical data must be screened against PSLs to determine if laboratory quantitation limits were adequate (However, in order to resolve the project problems and make decisions, separate screening levels are described in Section 11.4). For this project, there are PSLs for surface soil, subsurface soil, sediment, and groundwater. These PSLs are identified on Worksheet #15 and were selected using the following rationales:

- **Surface soil PSLs** – The PSLs are the lowest of the applicable human health risk-screening criteria, the RIDEM residential direct exposure criteria (RDEC), the RIDEM leachability criteria, and the selected ecological soil screening levels (SSLs), for the receptors identified in Section 10.5.
- **Subsurface soil PSLs** – Ecological risk is only applicable for surface soil. Therefore, the PSLs are the lowest of the same risk-screening criteria as for Category 1 surface soil, excluding the ecological SSLs.
- **Sediment PSLs** – The PSLs for sediment are the lowest of the applicable human health risk-screening criteria and the selected ecological sediment screening levels (SLs), for the receptors identified in Section 10.5.
- **Groundwater PSLs –** The PSLs are the lowest of the EPAs enforceable drinking water standards (Maximum Contaminant Levels [MCLs]), the RIDEM GA groundwater objectives, the human health risk-screening criteria, or the EPAs vapor intrusion (VI) guidance values.

(Note: PSLs are subject to change, based on ongoing research, and updated values will be used when screening is performed. PSLs that are current at the time of the risk screening will be used.)

Fixed laboratory analytical methods must be selected such that the subcontracted laboratories can achieve limits of quantitation (LOQs) less than or equal to the PSLs, to the extent technically feasible using conventional methods. To simplify the sampling and analysis procedures, the lowest of the surface and subsurface PSLs for each analyte was designated as the “soil PSL,” and method selection was

based on this lowest value for all of the types of soil. Worksheets #15a, #15b, and #15c present the PSLs; the selected methods; and the laboratory LOQs, limits of detection (LODs), and detection limits (DLs) for each analyte, for soil, and for groundwater, respectively.

The laboratories will measure concentrations of analytes, except dioxins, down to the laboratory DL, and of dioxins down to the sample-specific estimated detection limit (EDL). Positive detections of analytes, except dioxins, between the LOQ and the DL, and of dioxins between the LOQ and the EDL, must be qualified as estimated "J." The "J" alerts the data user to the increased uncertainty at concentrations between the DL and LOQ. Use of J-flagged data to achieve project goals is acceptable; however, greater scrutiny must be applied to J-qualified data.

Non-detected results must be qualified as "U" and must be reported with an associated value of the LOD, except for dioxins. Non-detected dioxins results must be reported with an associated value of the EDL, as provided in the analytical method.

For the purpose of making the decisions identified in Section 11.4, non-detected results with associated values greater than the PSL will be treated as values that are less than the PSL if the chemical was not detected in site media during this investigation or in previous investigations; otherwise, such results will be assigned a value equal to one-half the LOD (or, for dioxins, one-half the EDL). The limitations on data usability due to unmet sensitivity goals will be evaluated as described in Worksheet #37 and discussed in the project report. The data usability assessment will consider uncertainty associated with LOQ and/or LOD and EDL values that are greater than the PSL, and will evaluate whether the inability to detect or quantify an analyte at levels equal to or less than the PSL creates a data gap that has an adverse effect on decision making.

The background data set for various media at NAVSTA Newport must be used to determine whether metals present onsite are naturally occurring or site-related. Background data are described in the "Basewide Background Study Report for Naval Station Newport, Newport Rhode Island" (Tetra Tech, July 2008).

11.3 STUDY BOUNDARIES

11.3.1 AOC 001 Boundaries

The areas of focus for AOC 001, where soil and sediment data potentially will be used for CERCLA-type risk assessment, is in areas where tank bottom sludge was presumably burned (Figure 2). Two general soil populations must be represented in order to resolve the Category 1 problems – soil that potentially contains chemicals related to sludge-burning at concentrations that exceed the PSLs and background concentrations, and soil that does not. Soil adjacent to the west and south sides of the structure were previously found to be contaminated with TPH. Since previous investigations of similar structures at Tank Farms 4 and 5 have indicated that TPH is generally a good indicator of contamination by other

constituents, soil from these same locations must be investigated. Soil samples must also be collected west and south of the previous samples, and shallow soil samples must be collected to determine if the fill above the contamination is impacted.

The soil depth intervals of interest are those set forth by USEPA policy for risk assessment to define surface soil and subsurface soil. Surface soil is defined as soil collected to 1 foot bgs. Subsurface soil is defined as soil collected between 1 and 10 feet bgs, or to top of bedrock, whichever is shallower.

Sediment data of primary importance must represent areas potentially impacted by the discharge from the sand filter/burn chamber. In addition, sediment data representing areas not potentially contaminated as a result of site operations must be collected as a point of reference for evaluating the Site data. Previous investigations at the Site have indicated possible impacts to Lawton Brook from non-Site sources; therefore, areas near sampling locations SD-01 and SD-02 (Figure 2) upstream from any potential impacts from AOC 001 are appropriate to provide reference/upstream locations to assist in quantification of potential sediment impacts from areas other than the areas being investigated in this data gaps assessment (DGA). Sediment sample locations shown on Figures 2 and 3 are approximate and must be confirmed in the field by selecting areas of sediment deposition. Sediment sample depths must be from 0 to 6-inches bgs, in order to collect samples from the zone of bioturbation. In addition, deeper sediment/ soil must be collected to determine the depth of contamination, if present. Therefore, in addition to the collection of sediment samples from the 0-6 inch interval, soil borings will be advanced using a hand auger at sediment stations SD03, SD04 and SD05. Borings will be advanced to a maximum of 5 ft bgs or refusal and one subsurface sample will be selected from one of these deeper intervals. The sample interval will be one foot and the depth will be chosen based on visual or olfactory evidence, or biased towards fine grained material if no visual or olfactory evidence of contamination is found.

Contaminants from sludge-burning in AOC 001 areas do not appear to have impacted groundwater because previous groundwater sampling down-gradient of AOC 001 does not indicate contamination. However, shallow groundwater must be investigated down-gradient of the burn pit to confirm that contaminants have not reached the water table. The area is small, so a limited amount of data is anticipated to be sufficient.

Surface water has previously been investigated, and does not appear to have been impacted by Site operations; therefore, surface water is not within the scope of this investigation.

11.3.2 AOC 020

The areas of focus for AOC 020 are the areas around the pad-mounted transformers, where PCB contamination was previously detected (three soil samples). PCBs in surface soil were previously detected at the north side of transformer 1 and the east and west sides of transformer 2 (Figure 5). These three surface sample locations must be re-sampled as they provide a high degree of probability for detecting site-related contamination. Subsurface soil data must be collected from the same location to

support an evaluation of the potential for vertical migration of PCBs. In addition, soil approximately 10 to 15 feet away from each of the three previous soil samples must be investigated to serve as an initial attempt at delineating contamination in lateral directions.

The soil depth intervals of interest are those set forth by USEPA policy for risk assessment to define surface soil and subsurface soil. Surface soil is be defined as soil collected to 1 foot bgs. Subsurface soil is defined as soil collected between 1 and 10 feet bgs, or to top of bedrock, whichever is shallower.

Contaminants from the transformers in AOC 020 areas do not appear to have impacted groundwater because previous groundwater sampling down-gradient of AOC 020 does not indicate contamination. However, groundwater data must be collected from the shallow water bearing zone down-gradient of the transformers to establish whether contaminants have reached the water table.

11.3.3 Building 227

The areas of focus for the ECH/Building 227 are each side of Building 227, each entrance to Building 227, and the pipe from the south side of Building 227 (Figure 4). These are the areas representing the greatest chance for finding site-related contamination. Previous sampling was only performed for TPH around the pipe on the south side of Building 227.

The soil depth intervals of interest are those set forth by USEPA policy for risk assessment to define surface soil and subsurface soil. Surface soil is defined as soil collected to 1 foot bgs. Subsurface soil is defined as soil collected between 1 and 10 feet bgs, or to top of bedrock, whichever is shallower.

If contaminants from the transformer and possible batteries in Building 227 are present in soil, they may have impacted groundwater. To determine if this has occurred, groundwater samples will be collected for analysis of target analyte concentrations in existing monitoring well GZ-318. In addition a new groundwater monitoring well (TF3-ECH-MW01) will be installed.

11.4 DEVELOP THE ANALYTIC APPROACH

The rules described in this section will be used to evaluate the newly-acquired and usable historical chemical data and to make decisions regarding the problems described in Section 11.1.

11.4.1 Decision Rules

The following rule applies to decisions regarding Problem statements for all three Category 1 areas:

- If all measured concentrations in all surface and subsurface soil samples, sediment samples (if applicable), and the groundwater sample collected from a targeted Category 1 area are less than background concentrations (see Section 11.4.3) and less than the screening levels below, then the risk evaluation and delineation of contamination are complete and there is no unacceptable risk from the area. In this case, present the data and the risk evaluation in a DGA report; otherwise convene

the Project Team to evaluate an appropriate course of action. It must be noted that screening levels for the decision rules described in this section are different than PSLs described in Section 11.2.3 (which are used to set desired laboratory quantitation limits). Screening levels are defined as follows.

- Soil screening level: the lowest of the applicable human health risk-screening criteria, and the selected ecological soil screening levels (SSLs), (for surface soil only).
- Groundwater screening level: the lowest of the EPAs enforceable drinking water standards (MCLs), the human health risk-screening criteria, or the EPAs vapor intrusion (VI) guidance values.
- Sediment screening level: the lowest of the applicable human health risk-screening criteria and the selected ecological sediment screening levels (SLs), for the receptors identified in Section 10.5.

In the case of dioxins and furans, the analytical results will be compared to the total toxicity equivalency (TEQ) of 2,3,7,8-TCDD. The measured concentrations to be compared with the screening levels will be the total TEQs, calculated by multiplying each dioxin and furan congener concentration by that congener's toxicity equivalency factor (TEF) and summing the results. The total TEQ screening levels are the same as the PSLs presented in Worksheets #15a and #15c for 2,3,7,8-TCDD, which has a TEF of 1. (The individual congener PSLs presented in Worksheets #15a and #15c will not be compared with individual congener concentrations to make project decisions in accordance with this decision rule. These individual PSLs are presented to provide approximate values for the evaluation of analytical sensitivity only).

To implement this rule, existing data will be combined with newly collected data and together will be evaluated for various characteristics. During this evaluation, the Project Team will consider the following factors (see note below) as they relate to the individual AOCs (refer to Sections 11.1 and 11.2):

- Magnitude of screening level (or MCL) exceedances for the targeted analytes.
- Observed concentration gradients.
- Groundwater flow directions.
- The number of screening level exceedances.
- An evaluation of Site data compared to reference data (e.g. upgradient sediment data) to estimate whether contaminants in the affected environmental media appear to be related to Site operations or are associated with another source of contamination (See Section 11.4.2).
- Other factors the Project Team considers to be important for evaluating risks to human health (soil sediment and groundwater data) and ecological receptors (surface soil and sediment data only).

Note: The data and the risk screening (comparison of site target analyte concentrations to screening levels) will be presented in a DGA report. The DGA report will be prepared with figures and tables,

including the presentation of all exceedances of the applicable human health and ecological criteria identified in Section 11.2.3, and a meeting will be convened with the Project Team to discuss the next steps to be taken.

The tendency will be to act quickly to reduce risks if an immediate or near term threat to human health or the environment is identified. This condition would generally be indicated by high target analyte concentrations over large areas and may be followed, for example, by an Engineering Evaluation/Cost Analysis, an Interim Action, etc., designed to quickly evaluate and perhaps implement methods of reducing risks.

The tendency will be to recommend additional data collection if the project Team determines that contamination is insufficiently delineated or otherwise insufficiently characterized to support a risk screening and, if a risk screening indicates that risks may be unacceptable, a risk assessment. This condition would generally be indicated by not collecting the targeted data, or determining that the collected data do not adequately represent potential risks incurred by human or ecological receptors. This is most likely to exist for Building 227, where this project primarily requires a determination of presence/absence of contamination rather than full delineation of the contamination (See Section 11.1, Building 227).

11.4.2 Background Comparisons

Comparisons to background soil concentrations will be used to evaluate metals contamination. Metals commonly occur due to their presence in soil, attributable to geologic conditions. PAHs and dioxins are ubiquitous due to atmospheric deposition from human activities and/or natural sources (forest fires).

The background dataset for metals is in the Basewide Background Study Report for Naval Station Newport (Background Study). The method used for comparison between datasets for metals is outlined in the Background Study. For metals, when the soil type present at the site can be determined or matched to a particular soil type considered in the background study, a standard comparison can be made using 95 percent upper confidence level (UCL) of the two data sets. The geochemical method will be used as outlined in the Background Study when the soil type present is unknown or cannot be matched to a particular soil type considered in the Background Study.

11.5 SPECIFY PERFORMANCE CRITERIA

The sample locations were selected based on the need to characterize the nature and extent of contamination at the Site. Although this investigation emphasizes detection of contamination, to the extent practicable, the soil and sediment analytical data will be used to map the spatial boundaries of soil and sediment containing contaminant concentrations exceeding screening levels defined in Section 11.4.1. Particular scrutiny will be applied to analytical results below the LOQ when screening levels are below the LOQ. The data usability evaluation process is described in more detail in Worksheet #37.

The data collected under this SAP are anticipated to be sufficient to delineate the nature and extent of contamination in soil and sediment and support potential baseline risk assessments for the Site. The groundwater data is anticipated to be sufficient to determine if the three Category 1 areas have impacted groundwater. The project team will review the data as part of the data usability assessment described in Worksheet #37. If any significant data gaps are identified, the Project Team will determine the next appropriate step.

11.6 DATA COLLECTION PLAN

The plan for data collection is provided in detail in Worksheet #17.

SAP Worksheet #12 -- Measurement Performance Criteria Table (note matrix in table entry)
(UFP-QAPP Manual Section 2.6.2)

Measurement Performance Criteria Table – Field QC Samples(1)

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Trip Blank	VOCs, 1,2-Dibromoethane (EDB), GRO	One each per cooler, as appropriate	Accuracy /Bias/ Contamination	No target analytes > ½ LOQ (>LOQ for common laboratory contaminants), unless target analytes in field samples are > 10x those in trip blank.	S & A
Equipment Rinsate Blank(2)	All analytical groups	One per 20 samples	Accuracy /Bias/ Contamination	No target analytes > ½ LOQ (>LOQ for common laboratory contaminants), unless target analytes in field samples are > 10x those in rinsate blank.	S & A
Field Duplicates	Organics	One per 10 samples	Precision	Soils: Relative percent difference (RPD) must be ≤ 50%. Waters: RPD must be ≤ 30%. If sample results are < 2x LOQ, professional judgment is used.	S & A
	Metals	One per 10 samples	Precision	For values ≥ 5x LOQ Soils: RPD must be ≤ 50% Waters: RPD must be ≤ 30%. For values < 5x LOQ Soils: Absolute difference must be ≤ 4x LOQ Waters: Absolute difference must be ≤ 2x LOQ for waters.	S & A
Temperature Blank	All analytical groups(3)	One per cooler	Representativeness	Temperature must be ≤ 6 degrees Celsius (^{o}C)	S

1. The measurement performance criteria (MPCs) for laboratory QC samples are presented in Worksheet #28.
2. Equipment rinsate blanks will be collected if non-dedicated sampling equipment is used.
3. For metals, the MPC is only applicable for mercury in solid samples.

SAP Worksheet #13 -- Secondary Data Criteria and Limitations Table

(UFP-QAPP Manual Section 2.7)

Secondary Data	Data Source	Data Generator(s)	How Data Will Be Used	Limitations on Data Use
Report	Draft Tank Closure Assessment Report, Tank Farm 3. September 1998	GZA GeoEnvironmental, Inc.	Boring logs will be used when interpreting geologic and hydrogeologic data for the site. Some soil and groundwater data will be used for determinations of nature and extent of contamination.	Analyte lists differ between existing and new data, the existing data may only be useful for semiquantitative or qualitative interpretations of contaminant patterns. The older data was not validated and will not support a risk assessment.
Report	Supplemental Site Investigation and Corrective Action Plan Tank Farm 3. February, 1998.	GZA GeoEnvironmental, Inc.	Boring logs will be used when interpreting geologic and hydrogeologic data for the site. Some soil and groundwater data will be used for determinations of nature and extent of contamination.	Analyte lists differ between existing and new data, the existing data may only be useful for semiquantitative or qualitative interpretations of contaminant patterns. The older data was not validated and will not support a risk assessment.
Report	Draft SIRAR for Tank Farm 3. May, 2005.	Tetra Tech EC, Inc.	Some soil and groundwater data will be used for determinations of nature and extent of contamination.	Analyte lists differ between existing and new data, the existing data may only be useful for semiquantitative or qualitative interpretations of contaminant patterns. The older data was not validated and will not support a risk assessment.
Report	Supplemental Site Investigation Defense Fuel Supply Center Melville –Tank Farm 3	GZA GeoEnvironmental, Inc.	Boring logs will be used when interpreting geologic and hydrogeologic data for the site. Some soil and groundwater data will be used for determinations of nature and extent of contamination.	Analyte lists differ between existing and new data, the existing data may only be useful for semiquantitative or qualitative interpretations of contaminant patterns. The older data was not validated and will not completely support a risk assessment.

Secondary Data	Data Source	Data Generator(s)	How Data Will Be Used	Limitations on Data Use
Report	Basewide Background Investigation Report, Naval Station Newport, Newport Rhode Island. July 2008.	Tetra Tech Inc./ Metals	Data will be used to determine if metals present onsite are naturally occurring or are a result of historic site activities.	No limitations are applicable.

SAP Worksheet #14 -- Summary of Project Tasks
(UFP-QAPP Manual Section 2.8.1)

The following project tasks are summarized in the sections below:

- Field Tasks.
- Analytical Tasks.
- Data Management and Review.
- Project Report.

The Tetra Tech and USEPA standard operating procedures (SOPs) and field documentation forms referred to in this worksheet are included in Appendix B and Appendix C, respectively. Project-specific procedures for select field tasks are also provided in Appendix D. The field team will follow the project-specific field procedures unless these procedures do not provide guidance on a specific field task issue. In that case, the procedures in the cited SOPs will be followed.

14.1 FIELD TASKS

This project includes the following field tasks:

- Mobilization/Demobilization and Utility Clearance – includes mobilization of equipment and staff to the site, field team orientation, a site walkover, utility clearance, and demobilization. A DIGSAFE number and NAVSTA Newport utility clearance will be obtained prior to mobilizing drilling equipment. The site walk to pre-mark sampling locations will be held and regulatory personnel (EPA and RIDEM) will be invited to obtain concurrence of sample locations.
- Geophysical Survey – The discharge pipeline from the former sand filter (AOC 001) will be traced using geophysical techniques, to determine its location. As part of this effort, a video inspection of the pipe will also be conducted to locate any potential leaks or compromised areas. SOPs for geophysical techniques that may be used for tracing the discharge line are provided in Appendix B.
- Drilling and Soil Sample Collection – Soil borings will be advanced for continuous soil sampling using drilling methods described in SOP GH-1.3. Boring logs will be created according to SOP GH-1.5. Surface and subsurface soil samples for laboratory analysis will be collected from the borings according to SOP SA-1.3. Project-specific procedures for drilling and soil sampling are presented in Appendix D. The soil samples will be collected from the vadose zone at the intervals listed in Worksheet #18.
- Well installation, development and water level measurement – One new monitoring well will be installed and developed for groundwater sampling, in accordance with SOP GH-2.8. Three existing

monitoring wells will be developed for groundwater sampling. Existing monitoring wells will be evaluated, and water level measurements will be taken in all monitoring wells where samples will be taken and in two additional existing monitoring wells in the area of the electrical control house (ECH) and at AOC-001in accordance with SOP GH-1.2. Project specific procedures for drilling, well installation, development and evaluation; and water level measurements are presented in Appendix D. Groundwater flow direction will be determined and groundwater contour maps will be constructed, as described in SOP GH-2.5.

- Groundwater sampling – Groundwater samples will be collected following the USEPA's low flow (low stress) sampling protocol, SOP GW 001. Project-specific procedures are presented in Appendix D.
- Sediment sampling – Sediment samples will be collected using the project specific procedures presented in Appendix D and in accordance with SOP #SA 1.2.SW.
- Field Quality Control Samples – Field quality control (QC) samples will be collected as part of the investigation, including field duplicates, trip blanks, and equipment rinsate blanks. Worksheet #20 presents the field QC sample summary.

  Field samples to be used for laboratory QC analyses will be assigned by the field sampler on the chain-of-custody form and sample log sheet. The laboratory will perform matrix spike (MS) and matrix spike duplicate (MSD) analyses for organic analyses and MS and laboratory duplicate analyses for metals analysis. Additional sample volume will be collected as necessary for the laboratory QC analyses.
- Field Instrument Calibration – These procedures are described in Worksheet #22.
- Equipment decontamination – All non-disposable equipment that comes in contact with the sample medium will be decontaminated according to SOP SA-7.1 to prevent cross-contamination between sampling points. This includes equipment such as stainless steel bowls, scoops, as well as heavy equipment. Personnel decontamination is discussed in the HASP.

  All heavy equipment, including the drilling rig, rods and augers, and other down-hole equipment used during site investigation activities, will be decontaminated prior to beginning work and between all boreholes using a high-pressure steam wash. Potable water will be used for steam-cleaning.
- Investigation-Derived Waste (IDW) Characterization and Disposal – IDW includes decontamination fluid, used personal protective equipment (PPE), used sampling equipment, and drill cuttings and excess soil samples. IDW characterization and disposal will be performed after all IDW has been containerized. IDW shall be handled in accordance with SOP SA-7.1.
- Land Surveying – After completion of sample collection, the coordinates of all sample points, including soil borings, monitoring wells, as well as other pertinent features will be determined by a

Rhode Island registered land surveyor. The coordinates of the features will be incorporated into the NAVSTA Newport GIS database and used for site mapping.

14.2 ANALYTICAL TASKS

Chemical analysis of soil samples will be performed by subcontracted laboratories. TestAmerica will perform the dioxin analysis and Katahdin will perform VOC, PAH, PCBs, metals and TPH (GRO and Extractable Total Petroleum Hydrocarbons [ExTPH]) analyses in accordance with the methods identified in Worksheet #19 and the requirements of the analytical specifications for laboratory services developed for this work by Tetra Tech.

TestAmerica and Katahdin will follow the laboratory-specific SOPs (Worksheets #19 and #23) developed, based on the methods listed in Worksheet #19. Copies of the Laboratory SOPs are included in Appendix E.

All soil and sediment sample analytical results will be reported by the laboratory on a dry-weight basis. Results of percent moisture will be reported in each analytical data package and electronic data files. This information will also be captured in the project database, which will eventually be uploaded to the Naval Installation Restoration Information Solutions (NIRIS) database. Percent moisture information will also be captured in the DGA Report.

The analytical data packages provided by TestAmerica and Katahdin will be in a USEPA Contract Laboratory Program-like format, will be fully validatable, and contain raw data summary forms for all sample and laboratory method blank data, and summary forms containing all method specific quality control (results, recoveries, relative percent differences, relative standard deviations, and/or percent differences, etc.)

Results will be reported in each analytical data package and electronic data deliverable (EDD). This information will also be captured in the project database that will eventually be uploaded to the NIRIS.

14.3 DATA MANAGEMENT

Data management will be performed in accordance with SOP CT-05. Data management procedures will include the following:

- **Project documentation and records**
  - Field sample collection and field measurement records are described in Worksheets #27 and #29.
  - Laboratory data package deliverables are described in the analytical specifications.

- Data assessment documents and records are listed in Worksheet #29.

- **Data recording formats** are described in Worksheet #27.

- **Data handling and management** - After the field investigation is completed, the field sampling log sheets will be organized by date and media and filed in the project files. The field logbooks for this project will be used only for this Site, and will also be categorized and maintained in the project files after the completion of the field program. Project personnel completing concurrent field activities may maintain multiple field logbooks. When possible, logbooks will be segregated by sampling activity. The field logbooks will be titled based on date and activity. The data handling procedures to be followed by the laboratories will meet the requirements of the technical specification. The electronic data results will be automatically downloaded into the Tetra Tech database, in accordance with proprietary Tetra Tech processes.

- **Data tracking and control** - The Tetra Tech PM (or designee) is responsible for the overall tracking and control of data generated for the project.

  - **Data Tracking.** Data is tracked from its generation to its archiving in the Tetra Tech project-specific files. The Project Chemist (or designee) is responsible for tracking the samples collected and shipped to the contracted laboratory. On receipt of the data packages from the analytical laboratory, the Project Chemist will oversee the data validation effort, which includes verifying that the data packages are complete and that results for all samples have been delivered by the analytical laboratory.

  - **Data Storage, Archiving, and Retrieval.** The data packages received from the subcontract laboratory are tracked in the data validation log book. After the data are validated, the data packages are entered into the Tetra Tech CLEAN file system and archived in secure files. The field records including field logbooks, sample logs, chain-of-custody records, and field calibration logs will be submitted by the FOL to be entered into the CLEAN file system prior to archiving in secure project files. The project files are audited for accuracy and completeness. At the completion of the Navy contract, the records will be stored by Tetra Tech.

- **Data Security.** The Tetra Tech project files are restricted to designated personnel only. Records can only be borrowed temporarily from the project file using a sign-out system. The Tetra Tech Data Manager maintains the electronic data files. Access to the data files is restricted to qualified personnel only. File and data backup procedures are routinely performed.

14.4 DATA REVIEW

Data review is described in other worksheets, as follows:

- Data verification is described in Worksheet #34.

- Data validation is described in Worksheets #35 and #36.
- Usability assessment is described in Worksheet #37.

14.5 PROJECT REPORT

Following completion of the investigations outlined in this SAP, the Navy will prepare a Draft DGA Report, in accordance with the decision rules in Worksheet #11.4. This document will summarize the investigation activities; describe any issues encountered in the field and corrective actions taken; provide tables comparing soil and groundwater sampling results to screening criteria, defined in Section 11.4.1; and provide figures depicting the locations sampled and the spatial distribution of contaminants. The Draft Technical Report will also contain recommendations for the next steps for the Site.

The Draft Technical Report will be submitted to RIDEM and the USEPA for review. On receipt of regulatory comments, a response will be prepared, and if warranted, a meeting or conference call will be held to resolve comments. A Final Technical Report incorporating comments will be issued for inclusion in the NAVSTA Newport Administrative Record.

SAP Worksheet #15a – Reference Limits and Evaluation Table
(UFP-QAPP Manual Section 2.8.1)

In Worksheets #15a and #15b, the PSL is presented in bold font if it is less than the LOQ but greater than or equal to the LOD; and the PSL is presented as bolded and shaded if it is less than the LOD. The limitations on data usability due to unmet sensitivity goals will be evaluated as described in Worksheet #37 and discussed in the project report.

Matrix: Soil

Analyte	CAS Number	Soil PSL[1] (mg/kg)	Soil PSL Reference[2]	Project LOQ Goal (mg/kg)	Katahdin Limits		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
VOCs by SW-846 8260B							
1,2,4-Trimethylbenzene	95-63-6	6.2	Res RSL	2.1	0.005	0.0025	0.00079
1,2-Dibromoethane (EDB)	106-93-4	**0.0001**	So/Air SSL	0.000033	0.005	0.0025	0.0012
1,3,5-Trimethylbenzene	108-67-8	7.2	So/Air SSL	2.4	0.005	0.0025	0.00067
2-Butanone (MEK)	78-93-3	89.6	Eco SSL	30	0.025	0.0125	0.0059
2-Hexanone	591-78-6	12.6	Eco SSL	4.2	0.025	0.0125	0.0048
4-Methyl-2-pentanone (MIBK)	108-10-1	443	Eco SSL	150	0.025	0.0125	0.0059
Acetone	67-64-1	2.5	Eco SSL	0.83	0.025	0.0125	0.0051
Benzene	71-43-2	0.2	RIDEM GA LC	0.67	0.005	0.0025	0.00092
Bromoform	75-25-2	15.9	Eco SSL	5.3	0.005	0.0025	0.0007
Bromomethane	74-83-9	0.235	Eco SSL	0.078	0.01	0.005	0.0011
Carbon disulfide	75-15-0	0.0941	Eco SSL	0.031	0.005	0.0025	0.00078
Cyclohexane	110-82-7	700	Res RSL	230	0.005	0.0025	0.0014
Ethylbenzene	100-41-4	5.16	Eco SSL	1.7	0.005	0.0025	0.00065
Isopropylbenzene	98-82-8	27	RIDEM Res DEC	9	0.005	0.0025	0.00092
m,p-Xylenes[4]	179601-23-1	95	Eco SSL	32	0.01	0.005	0.0017
Methyl acetate	79-20-9	7800	Res RSL	2600	0.005	0.0025	0.0027
Methylcyclohexane	108-87-2	490	So/Air SSL	160	0.005	0.0025	0.00096
Methyl-tert-butyl ether	1634-04-4	43	Res RSL	1.3	0.005	0.0025	0.0011
Naphthalene	91-20-3	0.8	RIDEM Res DEC	0.27	0.005	0.0025	0.00088
n-Butylbenzene	104-51-8	--	--	--	0.005	0.0025	0.00092
n-Propylbenzene	103-65-1	340	Res RSL	11	0.005	0.0025	0.00083
o-Xylene[4]	95-47-6	95	Eco SSL	32	0.005	0.0025	0.0013

Matrix: Soil

Analyte	CAS Number	Soil PSL[1] (mg/kg)	Soil PSL Reference[2]	Project LOQ Goal (mg/kg)	Katahdin Limits		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
p-Isopropyltoluene	99-87-6	--	--	--	0.005	0.0025	0.00076
sec-Butylbenzene	135-98-8	--	--	--	0.005	0.0025	0.00091
Styrene	100-42-5	2.9	RIDEM GA LC	0.97	0.005	0.0025	0.00051
tert-Butylbenzene	98-06-6	--	--	--	0.005	0.0025	0.0009
Toluene	108-88-3	5.45	Eco SSL	1.8	0.005	0.0025	0.0014
Xylenes (total)	1330-20-7	63	Res RSL	21	0.015	0.0075	0.0013
PAHs by SW-846 8270D Selected Ion Monitoring (SIM)							
2-Methylnaphthalene	91-57-6	29	Eco SSL	9.7	0.02	0.01	0.0022
Acenaphthene	83-32-9	20	Eco SSL	6.7	0.02	0.01	0.0015
Acenaphthylene	208-96-8	23	RIDEM Res DEC	7.7	0.02	0.01	0.0012
Anthracene	120-12-7	29	Eco SSL	9.7	0.02	0.01	0.0012
Benzo(a)anthracene	56-55-3	0.15	Res RSL	0.05	0.02	0.01	0.0019
Benzo(a)pyrene	50-32-8	**0.015**	Res RSL	0.005	0.02	0.01	0.0033
Benzo(b)fluoranthene	205-99-2	0.15	Res RSL	0.05	0.02	0.01	0.0024
Benzo(g,h,i)perylene	191-24-2	0.8	RIDEM Res DEC	0.27	0.02	0.01	0.002
Benzo(k)fluoranthene	207-08-9	0.9	RIDEM Res DEC	0.3	0.02	0.01	0.0031
Chrysene	218-01-9	0.4	RIDEM Res DEC	0.13	0.02	0.01	0.0017
Dibenzo(a,h)anthracene	53-70-3	**0.015**	Res RSL	0.005	0.02	0.01	0.0018
Fluoranthene	206-44-0	20	RIDEM Res DEC	6.7	0.02	0.01	0.0018
Fluorene	86-73-7	28	RIDEM Res DEC	9.3	0.02	0.01	0.0032
Indeno(1,2,3-c,d)pyrene	193-39-5	0.15	Res RSL	0.05	0.02	0.01	0.0019
Naphthalene	91-20-3	0.8	RIDEM GA LC	0.27	0.02	0.01	0.0026
Phenanthrene	85-01-8	29	Eco SSL	9.7	0.02	0.01	0.0018
Pyrene	129-00-0	1.1	Eco SSL	0.37	0.02	0.01	0.0021
PCBs by SW-846 8082A							
Aroclor-1016	12674-11-2	0.39	Res RSL	0.13	0.017	0.0085	0.0060
Aroclor-1221	11104-28-2	0.14	Res RSL	0.047	0.017	0.0085	0.0079
Aroclor-1232	11141-16-5	0.14	Res RSL	0.047	0.017	0.010	0.0093
Aroclor-1242	53469-21-9	0.22	Res RSL	0.073	0.017	0.0085	0.0058
Aroclor-1248	12672-29-6	0.22	Res RSL	0.073	0.017	0.0085	0.0061
Aroclor-1254[5]	11097-69-1	0.11	Res RSL	0.037	0.017	0.0085	0.0047

Matrix: Soil

Analyte	CAS Number	Soil PSL[1] (mg/kg)	Soil PSL Reference[2]	Project LOQ Goal (mg/kg)	Katahdin Limits		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Aroclor-1260	11096-82-5	0.22	Res RSL	0.073	0.017	0.0085	0.0060
Aroclor-1262 [6]	37324-23-5	0.22	Res RSL	0.073	0.017	0.0085	0.0025
Aroclor-1268[6]	11100-14-4	0.22	Res RSL	0.073	0.017	0.0085	0.0025
Metals by SW-846 6020A[3]/7471B							
Aluminum	7429-90-5	50	Eco SSL	17	30	4	0.51
Antimony	7440-36-0	0.27	Eco SSL	0.09	0.1	0.05	0.020
Arsenic	7440-38-2	**0.39**	Res RSL	0.13	0.5	0.4	0.15
Barium	7440-39-3	330	Eco SSL	110	0.2	0.1	0.037
Beryllium	7440-41-7	1.5	RIDEM Res DEC	0.5	0.1	0.02	0.0041
Cadmium	7440-43-9	0.36	Eco SSL	0.12	0.1	0.02	0.0076
Calcium	7440-70-2	--	--	--	10	8	3.8
Chromium[7]	7440-47-3	**0.29**	Res RSL	0.097	0.3	0.2	0.049
Cobalt	7440-48-4	2.3	Res RSL	0.77	0.1	0.03	0.0054
Copper	7440-50-8	28	Eco SSL	9.3	0.3	0.2	0.071
Iron[8]	7439-89-6	200	Eco SSL	67	10	6	2.40
Lead	7439-92-1	11	Eco SSL	3.7	0.1	0.05	0.070
Magnesium	7439-95-4	--	--	--	10	8	1.37
Manganese	7439-96-5	180	Res RSL	60	0.2	0.1	0.042
Mercury	7439-97-6	0.1	Eco SSL	0.033	0.033	0.017	0.0052
Nickel	7440-02-0	38	Eco SSL	13	0.2	0.12	0.026
Potassium	7440-09-7	--	--	--	100	40	4.6
Selenium	7782-49-2	0.52	Eco SSL	0.17	0.5	0.3	0.039
Silver	7440-22-4	4.2	Eco SSL	1.4	0.1	0.04	0.0066
Sodium	7440-23-5	--	--	--	100	40	2.6
Thallium	7440-28-0	**0.0569**	Eco SSL	0.019	0.1	0.04	0.0094
Vanadium	7440-62-2	2	Eco SSL	0.67	0.5	0.4	0.11
Zinc	7440-66-6	46	Eco SSL	15	1	0.8	0.13
Petroleum Hydrocarbons							
GRO (C5-C12)	--	--	--	--	2.5	2	2
ExTPH (C8-C44)	--	--	--	--	20	10	5.7
TPH	--	500	Res DEC	170	5	3.8	2.6

Matrix: Soil

Analyte	CAS Number	Soil PSL[1] (pg/g)	Soil PSL Reference[2]	LOQ Goal (pg/g)	TestAmerica Limits		
					LOQ (pg/g)	LOD (pg/g)	DLs[9] (pg/g)
Dioxins and Furansby SW-846 8290							
1,2,3,4,6,7,8,9-OCDD[10]	3268-87-9	14000	So/Air SSL	4700	10	1.5	EDL
1,2,3,4,6,7,8,9-OCDF[10]	39001-02-0	14000	So/Air SSL	4700	10	1.5	EDL
1,2,3,4,6,7,8-HPCDD[10]	35822-46-9	420	So/Air SSL	140	5	0.75	EDL
1,2,3,4,6,7,8-HPCDF[10]	67562-39-4	420	So/Air SSL	140	5	0.75	EDL
1,2,3,4,7,8,9-HPCDF[10]	55673-89-7	420	So/Air SSL	140	5	0.75	EDL
1,2,3,4,7,8-HXCDD[10]	39227-28-6	42	So/Air SSL	14	5	0.75	EDL
1,2,3,4,7,8-HXCDF[10]	70648-26-9	42	So/Air SSL	14	5	0.75	EDL
1,2,3,6,7,8-HXCDD[10]	57653-85-7	42	So/Air SSL	14	5	0.75	EDL
1,2,3,6,7,8-HXCDF[10]	57117-44-9	42	So/Air SSL	14	5	0.75	EDL
1,2,3,7,8,9-HXCDD[10]	19408-74-3	42	So/Air SSL	14	5	0.75	EDL
1,2,3,7,8,9-HXCDF[10]	72918-21-9	42	So/Air SSL	14	5	0.75	EDL
1,2,3,7,8-PECDD[10]	40321-76-4	**4.2**	So/Air SSL	1.4	5	0.75	EDL
1,2,3,7,8-PECDF[10]	57117-41-6	140	So/Air SSL	46.7	5	0.75	EDL
2,3,4,6,7,8-HXCDF[10]	60851-34-5	42	So/Air SSL	14	5	0.75	EDL
2,3,4,7,8-PECDF[10]	57117-31-4	14	So/Air SSL	4.7	5	0.75	EDL
2,3,7,8-TCDD[10]	1746-01-6	4.2	So/Air SSL	1.4	1	0.15	EDL
2,3,7,8-TCDF[10]	51207-31-9	42	So/Air SSL	14	1	0.15	EDL
TOTAL HPCDD	37871-00-4	--	--	--	--	--	--
TOTAL HPCDF	38998-75-3	--	--	--	--	--	--
TOTAL HXCDD	34465-46-8	--	--	--	--	--	--
TOTAL HXCDF	55684-94-1	--	--	--	--	--	--
TOTAL PECDD	36088-22-9	--	--	--	--	--	--
TOTAL PECDF	30402-15-4	--	--	--	--	--	--
TOTAL TCDD	41903-57-5	--	--	--	--	--	--
TOTAL TCDF	55722-27-5	--	--	--	--	--	--

Notes:

1. Although there are separate PSLs for surface and subsurface soil, a single soil PSL representing the lowest of these PSLs is presented here, and the LOQ goals and selected methods are the same for all soil samples, in order to simplify sampling and analysis procedures. The soil PSLs presented are the lowest of:

   - EPA Regional Screening Levels (RSLs) residential and industrial soil values (EPA, 2012)

- EPA Soil to Air Soil Screening Levels (SSLs) (EPA, 2010b)
- RIDEM Residential Direct Exposure Criteria (RIDEM, 2011)
- Selected ecological SSL (applicable only for surface soil PSLs)

One-tenth values are displayed for non-cancer RSLs and Soil to Air SSLs to correspond to a target hazard quotient of 0.1. The selected ecological SSLs are the lowest of the selected benchmarks for plants, invertebrates, and wildlife. The benchmarks were selected by order of preference according to the following hierarchy:

Order of preference for plants and invertebrates:

1. EPA Ecological SSLs (U.S. EPA, 2003-2008)

2a. Oak Ridge National Laboratory (ORNL) Plant Toxicological Benchmark (Efroymson, 1997a)

2b. ORNL Invertebrate Toxicological Benchmark (Efroymson, 1997b)

3. Canadian Council and Ministers of Environment (CCME) (CCME, 1997-2010)

4. Target values for soil remediation (MHSPE, 2000)

Order of preference for wildlife:

1. EPA Ecological SSLs (U.S. EPA, 2003-2008)

2. CCME (CCME, 2010)

3. EPA Region 5 Ecological Screening Levels (U.S. EPA, 2003).

2. PSL Reference Abbreviations:
   - Eco SSL = Selected ecological SSL
   - Res RSL = EPA RSL residential soil value
   - So/Air SSL = EPA Soil to Air SSL
   - Res DEC = RIDEM Residential Direct Exposure Criteria
3. The LOQs, LODs, and DLs presented for metals analyzed by Method 6020A reflect an assumed dilution factor of 5, which is typically required for solids analysis by this method. If the dilution factor is different, the values will be adjusted accordingly.
4. PSL value is for xylenes.
5. The 1/10th non-cancer (N) value is lower than the cancer (C) value; therefore, the 1/10th N value is presented.
6. PSL value is for Aroclor 1260.
7. PSL value is for hexavalent chromium.

8. Standard is from "Efroymson, R.Al, M.E. Will, and G. W. Suter II. 1997b. Toxicological Benchmarks for Contaminants of Potential Concerns for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision. Oak Ridge National Laboratory. November. ES/ER/TM-126/R2.", and is based upon toxic effects to invertebrates.

9. Estimated Detection Limit (EDL) - For each chemical not detected, an EDL is calculated. The sample-specific EDL is an estimate made by the laboratory of the concentration of a given chemical that would have to be present to produce a signal with a peak height of at least 2.5 times the background signal level. The estimate is specific to a particular analysis of the sample and will be affected by sample preparation factors such as sample size, percent solids, and dilution. Non-detected results will be reported with an associated value of the EDL, and results between the LOQ and EDL will be flagged as estimated "J". LODs, and shading of PSLs below LODs, are presented for informational purposes.

10. PSL value presented is the screening level for total TEQ of 2,3,7,8-TCDD (4.2 pg/g), divided by the congener's 2005 World Health Organization (WHO) TEF for humans and mammals (Van den Berg, et al, 2006). This value is presented as an approximate value by which to evaluate analytical sensitivity, but it will not be compared with the individual dioxin or furan congener's concentrations to make project decisions according to the decision rules described in Worksheet 11, Section 11.4. To make the project decisions, each congener concentration will be multiplied by the congener's TEF; the TEF-adjusted concentrations of all congeners will be summed to obtain the total TEQ, and the total TEQ will be compared with the total TEQ PSL.

SAP Worksheet #15b – Reference Limits and Evaluation Table
(UFP-QAPP Manual Section 2.8.1)

Matrix:
Groundwater

Analyte	CAS Number	Groundwater PSL[1] (µg/L)	Groundwater PSL Reference[1]	Project LOQ Goal (µg/L)	Katahdin Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
1,2-Dibromoethane (EDB) by SW-846 8011	106-93-4	**0.0065**	RSL Tapwater	0.0022	0.05	0.025	0.0073
VOCs by SW-846 8260B							
1,2,4-Trimethylbenzene	95-63-6	1.5	RSL Tapwater	0.5	1	0.5	0.19
1,3,5-Trimethylbenzene	108-67-8	8.7	RSL Tapwater	2.9	1	0.5	0.2
2-Butanone (MEK)	78-93-3	490	RSL Tapwater	160	5	2.5	1.31
2-Hexanone	591-78-6	**3.4**	RSL Tapwater	1.1	5	2.5	1.7
4-Methyl-2-pentanone (MIBK)	108-10-1	100	RSL Tapwater	67	5	2.5	1.32
Acetone	67-64-1	1200	RSL Tapwater	400	5	2.5	2.21
Benzene	71-43-2	**0.39**	RSL Tapwater	0.13	1	0.5	0.26
Bromoform	75-25-2	7.9	RSL Tapwater	0.0028	1	0.5	0.23
Bromomethane	74-83-9	**0.7**	RSL Tapwater	0.23	2	1	0.49
Carbon disulfide	75-15-0	72	RSL Tapwater	24	1	0.5	0.25
Cyclohexane	110-82-7	100	EPA VI Guidance	33	1	0.5	0.31
Ethylbenzene	100-41-4	1.3	RSL Tapwater	0.5	1	0.5	0.21
Isopropylbenzene	98-82-8	39	RSL Tapwater	13	1	0.5	0.23
m,p-Xylenes[2]	179601-23-1	19	RSL Tapwater	6.3	2	1	0.59
Methyl acetate	79-20-9	1600	RSL Tapwater	533	1	0.5	0.5
Methylcyclohexane	108-87-2	--	--	--	1	0.5	0.3
Methyl-tert-butyl ether	1634-04-4	12	RSL Tapwater	4	1	0.5	0.36
Naphthalene	91-20-3	**0.14**	RSL Tapwater	0.047	1	0.5	0.3
n-Butylbenzene	104-51-8	78	RSL Tapwater	26	1	0.5	0.23
n-Propylbenzene	103-65-1	53	RSL Tapwater	18	1	0.5	0.26

Matrix: Groundwater

Analyte	CAS Number	Groundwater PSL[1] (µg/L)	Groundwater PSL Reference[1]	Project LOQ Goal (µg/L)	Katahdin Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
o-Xylene	95-47-6	19	RSL Tapwater	6.3	1	0.5	0.25
p-Isopropyltoluene	99-87-6	--	--	--	1	0.5	0.25
sec-Butylbenzene[10]	135-98-8	78	RSL Tapwater	26	1	0.5	0.21
Styrene	100-42-5	110	EPA MCL/ RIDEM GA Objective	37	1	0.5	0.23
tert-Butylbenzene[10]	98-06-6	78	RSL Tapwater	26	1	0.5	0.31
Toluene	108-88-3	86	RSL Tapwater	29	1	0.5	0.27
Xylenes (total)	1330-20-7	19	RSL Tapwater	6.3	3	1.5	0.25
PAHs by SW-846 8270D SIM							
2-Methylnaphthalene	91-57-6	2.7	RSL Tapwater	0.9	0.2	0.1	0.077
Acenaphthene	83-32-9	40	RSL Tapwater	13	0.2	0.1	0.064
Acenaphthylene[3]	208-96-8	40	RSL Tapwater	13	0.2	0.1	0.054
Anthracene	120-12-7	130	RSL Tapwater	43	0.2	0.1	0.044
Benzo(a)anthracene	56-55-3	**0.029**	RSL Tapwater	0.0097	0.2	0.1	0.046
Benzo(a)pyrene	50-32-8	**0.0029**	RSL Tapwater	0.00097	0.2	0.1	0.066
Benzo(b)fluoranthene	205-99-2	**0.029**	RSL Tapwater	0.0097	0.2	0.1	0.089
Benzo(g,h,i)perylene[4]	191-24-2	8.7	RSL Tapwater	2.9	0.2	0.1	0.065
Benzo(k)fluoranthene	207-08-9	0.29	RSL Tapwater	0.097	0.2	0.1	0.049
Chrysene	218-01-9	2.9	RSL Tapwater	0.97	0.2	0.1	0.036
Dibenzo(a,h)anthracene	53-70-3	**0.0029**	RSL Tapwater	0.00097	0.2	0.1	0.07
Fluoranthene	206-44-0	63	RSL Tapwater	21	0.2	0.1	0.073
Fluorene	86-73-7	22	RSL Tapwater	7.3	0.2	0.1	0.061
Indeno(1,2,3-c,d)pyrene	193-39-5	**0.029**	RSL Tapwater	0.0097	0.2	0.1	0.052
Naphthalene	91-20-3	**0.14**	RSL Tapwater	0.047	0.2	0.1	0.064
Phenanthrene[4]	85-01-8	8.7	RSL Tapwater	2.9	0.2	0.1	0.051

Matrix:
Groundwater

Analyte	CAS Number	Groundwater PSL[1] (µg/L)	Groundwater PSL Reference[1]	Project LOQ Goal (µg/L)	Katahdin Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Pyrene	129-00-0	8.7	RSL Tapwater	2.9	0.2	0.1	0.059
PCBs by SW-846 8082A							
Aroclor-1016[5]	12674-11-2	**0.11**	RSL Tapwater	0.034	0.50	0.25	0.15
Aroclor-1221	11104-28-2	**0.004**	RSL Tapwater	0.00013	0.50	0.25	0.20
Aroclor-1232	11141-16-5	**0.004**	RSL Tapwater	0.00013	0.50	0.25	0.089
Aroclor-1242	53469-21-9	**0.034**	RSL Tapwater	0.011	0.50	0.25	0.18
Aroclor-1248	12672-29-6	**0.034**	RSL Tapwater	0.011	0.50	0.25	0.20
Aroclor-1254	11097-69-1	**0.034**	RSL Tapwater	0.011	0.50	0.25	0.082
Aroclor-1260	11096-82-5	**0.034**	RSL Tapwater	0.011	0.50	0.25	0.17
Aroclor-1262[6]	37324-23-5	**0.034**	RSL Tapwater	0.011	0.50	0.25	0.07
Aroclor-1268[6]	11100-14-4	**0.034**	RSL Tapwater	0.011	0.50	0.25	0.07
Metals by SW-846 6020A[7]/7470A							
Aluminum	7429-90-5	1600	RSL Tapwater	533	300	40	4.4
Antimony	7440-36-0	**0.6**	RSL Tapwater	0.2	1	0.5	0.054
Arsenic	7440-38-2	**0.045**	RSL Tapwater	0.015	5	4	2.2
Barium	7440-39-3	290	RSL Tapwater	97	2	1	0.27
Beryllium	7440-41-7	4	EPA MCL	1.3	1	0.2	0.034
Cadmium	7440-43-9	**0.69**	RSL Tapwater	0.23	1	0.2	0.030
Calcium	7440-70-2	--	--	--	100	80	20
Chromium[8]	7440-47-3	**0.031**	RSL Tapwater	0.010	3	2	0.22
Cobalt	7440-48-4	**0.47**	RSL Tapwater	0.16	1	0.3	0.060
Copper	7440-50-8	62	RSL Tapwater	21	3	2	0.18
Iron	7439-89-6	1100	RSL Tapwater	367	100	60	13
Lead	7439-92-1	15	EPA MCL	5	1	0.5	0.074
Magnesium	7439-95-4	--	--	--	100	80	7.8
Manganese	7439-96-5	**32**	RSL Tapwater	11	2	1	0.35
Mercury	7439-97-6	**0.067**	EPA VI Guidance	0.022	0.2	0.1	0.013
Nickel	7440-02-0	30	RSL Tapwater	10	2	1.2	0.151
Potassium	7440-09-7	--	--	--	1000	400	31
Selenium	7782-49-2	7.8	RSL Tapwater	2.6	5	3	0.19
Silver	7440-22-4	7.1	RSL Tapwater	2.4	1	0.4	0.050
Sodium	7440-23-5	--	--	--	1000	400	18

Matrix:
Groundwater

Analyte	CAS Number	Groundwater PSL[1] (µg/L)	Groundwater PSL Reference[1]	Project LOQ Goal (µg/L)	Katahdin Limits		
					LOQ (µg/L)	LOD (µg/L)	DL (µg/L)
Thallium	7440-28-0	2	EPA MCL/RIDEM GA Objective	0.67	1	0.4	0.060
Vanadium[9]	7440-62-2	7.8	RSL Tapwater	2.6	5	4	0.51
Zinc	7440-66-6	470	RSL Tapwater	157	10	8	3.9

Notes:
-- = Not available or not applicable

1. The groundwater PSL is the lowest of:
   - EPA RSL tapwater value (EPA, 2012) (RSL Tapwater)
   - EPA MCL (EPA, 2010a)
   - RIDEM GA Objective (RIDEM, 2011)
   - EPA's Vapor Intrusion Screening Level (VISL) Calculator Version 2.0 (EPA, May 2012). Values correspond to a target cancer risk level of 1E-6 and an attenuation factor of 0.001.

   One-tenth values are displayed for non-cancer RSLs and vapor intrusion (VI) values to correspond to a target HQ of 0.1.
2. PSL value is for m-xylene.
3. PSL value is for Acenaphthene.
4. PSL value is for pyrene.
5. The 1/10th non-cancer (N) value is lower than the cancer (C) value; therefore, the 1/10th N value is presented.
6. PSL value is for Aroclor 1260.
7. The LOQs, LODs, and DLs presented for metals analyzed by Method 6020A reflect an assumed dilution factor of 5, which is typically required for groundwater analysis by this method. If the dilution factor is different, the values will be adjusted accordingly.
8. PSL value is for hexavalent chromium.
9. PSL value is for vanadium and compounds.
10. PSL value is for n-Butylbenzene.

SAP Worksheet #15c – Reference Limits and Evaluation Table
(UFP-QAPP Manual Section 2.8.1)

Matrix: Sediment

Analyte	CAS Number	Sediment PSL[1] (mg/kg)	Sediment PSL Reference[2]	Project LOQ Goal (mg/kg)	Katahdin Limits		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
VOCs by SW-846 8260B							
1,2,4-Trimethylbenzene	95-63-6	6.2	RSL Res	2.1	0.005	0.0025	0.00087
1,2-Dibromoethane (EDB)	106-93-4	0.034	RSL Res	0.011	0.005	0.0025	0.0012
1,3,5-Trimethylbenzene	108-67-8	78	RSL Res	26	0.005	0.0025	0.00067
2-Butanone (MEK)	78-93-3	0.27	Eco Sed SL	0.09	0.025	0.0125	0.0059
2-Hexanone	591-78-6	**0.022**	Eco Sed SL	0.0073	0.025	0.0125	0.0048
4-Methyl-2-pentanone (MIBK)	108-10-1	0.033	Eco Sed SL	0.011	0.025	0.0125	0.0059
Acetone	67-64-1	**0.0087**	Eco Sed SL	0.0029	0.025	0.0125	0.0051
Benzene	71-43-2	0.16	Eco Sed SL	0.053	0.005	0.0025	0.00092
Bromoform	75-25-2	0.654	Eco Sed SL	0.22	0.005	0.0025	0.0007
Bromomethane	74-83-9	0.73	RSL Res	0.24	0.01	0.005	0.0011
Carbon disulfide	75-15-0	**0.000851**	Eco Sed SL	0.00028	0.005	0.0025	0.00078
Cyclohexane	110-82-7	700	RSL Res	230	0.005	0.0025	0.0014
Ethylbenzene	100-41-4	1.1	Eco Sed SL	0.37	0.005	0.0025	0.00065
Isopropylbenzene	98-82-8	0.08	Eco Sed SL	0.027	0.005	0.0025	0.00092
m,p-Xylenes[3]	179601-23-1	59	RSL Res	20	0.01	0.005	0.0017
Methyl acetate	79-20-9	7800	RSL Res	2600	0.005	0.0025	0.0027
Methylcyclohexane	108-87-2	--	--	--	0.005	0.0025	0.00096
Methyl-tert-butyl ether	1634-04-4	43	RSL Res	14	0.005	0.0025	0.0011
Naphthalene	91-20-3	0.176	Eco Sed SL	0.059	0.005	0.0025	0.00088
n-Butylbenzene	104-51-8	--	--	--	0.005	0.0025	0.00092
n-Propylbenzene	103-65-1	340	RSL Res	110	0.005	0.0025	0.00083
o-Xylene	95-47-6	69	RSL Res	23	0.005	0.0025	0.0013
p-Isopropyltoluene	99-87-6	--	--	--	0.005	0.0025	0.00076
sec-Butylbenzene	135-98-8	--	--	--	0.005	0.0025	0.00091
Styrene	100-42-5	0.559	Eco Sed SL	0.19	0.005	0.0025	0.00051
tert-Butylbenzene	98-06-6	--	--	--	0.005	0.0025	0.0009
Toluene	108-88-3	0.05	Eco Sed SL	0.017	0.005	0.0025	0.0014
Xylenes (total)	1330-20-7	0.16	Eco Sed SL	0.053	0.015	0.0075	0.0013
PAHs by SW-846 8270D SIM							
2-Methylnaphthalene	91-57-6	0.0202	Eco Sed SL	0.0067	0.02	0.01	0.0022
Acenaphthene	83-32-9	**0.0067**	Eco Sed SL	0.0022	0.02	0.01	0.0015

Matrix: Sediment

Analyte	CAS Number	Sediment PSL[1] (mg/kg)	Sediment PSL Reference[2]	Project LOQ Goal (mg/kg)	Katahdin Limits		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Acenaphthylene	208-96-8	**0.0059**	Eco Sed SL	0.002	0.02	0.01	0.0012
Anthracene	120-12-7	0.0572	Eco Sed SL	0.019	0.02	0.01	0.0012
Benzo(a)anthracene	56-55-3	0.108	Eco Sed SL	0.036	0.02	0.01	0.0019
Benzo(a)pyrene	50-32-8	**0.015**	RSL Res	0.005	0.02	0.01	0.0033
Benzo(b)fluoranthene[4]	205-99-2	0.13	Eco Sed SL	0.043	0.02	0.01	0.0024
Benzo(g,h,i)perylene	191-24-2	0.17	Eco Sed SL	0.057	0.02	0.01	0.002
Benzo(k)fluoranthene	207-08-9	0.24	Eco Sed SL	0.08	0.02	0.01	0.0031
Chrysene	218-01-9	0.166	Eco Sed SL	0.055	0.02	0.01	0.0017
Dibenzo(a,h)anthracene	53-70-3	**0.015**	RSL Res	0.005	0.02	0.01	0.0018
Fluoranthene	206-44-0	0.423	Eco Sed SL	0.14	0.02	0.01	0.0018
Fluorene	86-73-7	0.0774	Eco Sed SL	0.026	0.02	0.01	0.0032
Indeno(1,2,3-c,d)pyrene	193-39-5	**0.017**	Eco Sed SL	0.0057	0.02	0.01	0.0019
Naphthalene	91-20-3	0.176	Eco Sed SL	0.059	0.02	0.01	0.0026
Phenanthrene	85-01-8	0.204	Eco Sed SL	0.068	0.02	0.01	0.0018
Pyrene	129-00-0	0.195	Eco Sed SL	0.065	0.02	0.01	0.0021
Metals by SW-846 6020A[5]/7471B							
Aluminum	7429-90-5	7700	RSL Res	2600	30	4	0.51
Antimony	7440-36-0	2	Eco Sed SL	0.67	0.1	0.05	0.020
Arsenic	7440-38-2	**0.39**	RSL Res	0.13	0.5	0.4	0.15
Barium[4]	7440-39-3	48	Eco Sed SL	16	0.2	0.1	0.037
Beryllium	7440-41-7	16	RSL Res	5.3	0.1	0.02	0.0041
Cadmium	7440-43-9	0.99	Eco Sed SL	0.33	0.1	0.02	0.0076
Calcium	7440-70-2	--	--	--	10	8	3.8
Chromium[6]	7440-47-3	**0.29**	RSL Res	0.097	0.3	0.2	0.049
Cobalt	7440-48-4	2.3	RSL Res	0.77	0.1	0.03	0.0054
Copper	7440-50-8	31.6	Eco Sed SL	11	0.3	0.2	0.071
Iron	7439-89-6	5500	RSL Res	1800	10	6	2.40
Lead	7439-92-1	35.8	Eco Sed SL	12	0.1	0.05	0.070
Magnesium	7439-95-4	--	--	--	10	8	1.37
Manganese	7439-96-5	180	RSL Res	60	0.2	0.1	0.042
Mercury	7439-97-6	0.18	Eco Sed SL	0.06	0.033	0.017	0.0052
Nickel	7440-02-0	22.7	Eco Sed SL	7.6	0.2	0.12	0.026
Potassium	7440-09-7	--	--	--	100	40	4.6
Selenium	7782-49-2	2	Eco Sed SL	0.67	0.5	0.3	0.039
Silver	7440-22-4	1	Eco Sed SL	0.33	0.1	0.04	0.0064

Matrix: Sediment

Analyte	CAS Number	Sediment PSL[1] (mg/kg)	Sediment PSL Reference[2]	Project LOQ Goal (mg/kg)	Katahdin Limits		
					LOQ (mg/kg)	LOD (mg/kg)	DL (mg/kg)
Sodium	7440-23-5	--	--	--	100	40	2.6
Thallium	7440-28-0	**0.078**	RSL Res	0.026	0.1	0.04	0.0094
Vanadium[7]	7440-62-2	39	RSL Res	13	0.5	0.4	0.11
Zinc	7440-66-6	121	Eco Sed SL	40	1	0.8	0.13
1,2,3,4,6,7,8,9-OCDD[9, 10]	3268-87-9	8500	Eco Sed SL	2800	10	1.5	EDL
1,2,3,4,6,7,8,9-OCDF[9, 10]	39001-02-0	8500	Eco Sed SL	2800	10	1.5	EDL
1,2,3,4,6,7,8-HPCDD[10, 11]	35822-46-9	450	RSL Res	150	5	0.75	EDL
1,2,3,4,6,7,8-HPCDF[9, 10]	67562-39-4	85	Eco Sed SL	28	5	0.75	EDL
1,2,3,4,7,8,9-HPCDF[9, 10]	55673-89-7	85	Eco Sed SL	28	5	0.75	EDL
1,2,3,4,7,8-HXCDD[9, 10]	39227-28-6	**1.7**	Eco Sed SL	0.6	5	0.75	EDL
1,2,3,4,7,8-HXCDF[9, 10]	70648-26-9	8.5	Eco Sed SL	2.8	5	0.75	EDL
1,2,3,6,7,8-HXCDD[10, 11]	57653-85-7	45	RSL Res	15	5	0.75	EDL
1,2,3,6,7,8-HXCDF[9, 10]	57117-44-9	8.5	Eco Sed SL	2.8	5	0.75	EDL
1,2,3,7,8,9-HXCDD[10, 11]	19408-74-3	45	RSL Res	15	5	0.75	EDL
1,2,3,7,8,9-HXCDF[9, 10]	72918-21-9	8.5	Eco Sed SL	2.8	5	0.75	EDL
1,2,3,7,8-PECDD[9, 10]	40321-76-4	**0.85**	Eco Sed SL	0.28	5	0.75	EDL
1,2,3,7,8-PECDF[9, 10]	57117-41-6	17	Eco Sed SL	6	5	0.75	EDL
2,3,4,6,7,8-HXCDF[9, 10]	60851-34-5	8.5	Eco Sed SL	2.8	5	0.75	EDL
2,3,4,7,8-PECDF[9, 10]	57117-31-4	**1.7**	Eco Sed SL	0.6	5	0.75	EDL
2,3,7,8-TCDD[9, 10]	1746-01-6	**0.85**	Eco Sed SL	0.28	1	0.15	EDL
2,3,7,8-TCDF[9, 10]	51207-31-9	17	Eco Sed SL	6	1	0.15	EDL
TOTAL HPCDD	37871-00-4	--	--	--	--	--	--
TOTAL HPCDF	38998-75-3	--	--	--	--	--	--
TOTAL HXCDD	34465-46-8	--	--	--	--	--	--
TOTAL HXCDF	55684-94-1	--	--	--	--	--	--
TOTAL PECDD	36088-22-9	--	--	--	--	--	--
TOTAL PECDF	30402-15-4	--	--	--	--	--	--
TOTAL TCDD	41903-57-5	--	--	--	--	--	--
TOTAL TCDF	55722-27-5	--	--	--	--	--	--

Notes:
-- = Not available or not applicable

1. The sediment PSLs are the lower of:

   - EPA Regional Screening Level (RSL) residential and industrial soil values (EPA, 2012)
   - Selected ecological sediment SLs

   One-tenth values are displayed for non-cancer RSLs to correspond to a target hazard quotient (HQ) of 0.1.The selected ecological sediment SLs were selected by order of preference according to the following hierarchy:

   1. EPA Region 3 Biological Technical Assistance Group (BTAG) Freshwater Sediment Screening Benchmarks (U.S. EPA, 2006a)
   2. Secondary Chronic Value (SCV) (Jones, D.S., et al, 1997)
   3. EPA Sediment Quality Benchmarks (SQB) (U.S. EPA, 2006b) (Based on 1% Total Organic Carbon.)
   4. National Oceanic and Atmospheric Administration (NOAA) Screening Quick Reference Tables (SQuiRT) Sediment Benchmarks (Buchman, M. F., 2008) (Freshwater value unless otherwise noted).

2. PSL Reference Abbreviations:

   - Eco Sed SL
   - Res RSL = EPA RSL residential soil value

3. PSL value is for m-xylene.

4. Eco Sed SL is a saltwater (marine sediment) value.

5. The LOQs, LODs, and DLs presented for metals analyzed by Method 6020A reflect an assumed dilution factor of 5, which is typically required for solids analysis by this method. If the dilution factor is different, the values will be adjusted accordingly.

6. PSL value is for hexavalent chromium.

7. PSL value is for vanadium and compounds.

8. Estimated Detection Limit (EDL) - For each chemical not detected, an EDL is calculated. The sample-specific EDL is an estimate made by the laboratory of the concentration of a given chemical that would have to be present to produce a signal with a peak height of at least 2.5 times the background signal level. The estimate is specific to a particular analysis of the sample and will be affected by sample preparation factors such as sample size, percent solids, and dilution. Non-detected results will be reported with an associated value of the EDL, and results between the LOQ and EDL will be flagged as estimated "J". LODs, and shading of PSLs below LODs, are presented for informational purposes.

9. PSL value presented is the screening level for total TEQ of 2,3,7,8-TCDD (0.85 picogram per gram pg/g), divided by the congener's 1998 WHO TEF for fish (Van den Berg, et al, 1998).

10. The PSL value is presented as an approximate value by which to evaluate analytical sensitivity, but it will not be compared with the individual dioxin or furan congener's concentrations to make project decisions according to the decision rules described in Worksheet 11, Section 11.4. To make the project decisions, each congener concentration will be multiplied by the congener's TEF; the TEF-adjusted concentrations of all congeners will be summed to obtain the total TEQ, and the total TEQ will be compared with the total TEQ PSL.

11. PSL value presented is the screening level for total TEQ of 2,3,7,8-TCDD (4.5 pg/g), divided by the congener's 2005 WHO TEF for humans and mammals (Van den Berg, et al, 2006).

SAP Worksheet #16 -- Project Schedule / Timeline Table (optional format)

(UFP-QAPP Manual Section 2.8.2)

Activities	Organization	Dates (MM/DD/YY)		Deliverable	Deliverable Due Date
		Anticipated Date(s) of Initiation	Anticipated Date of Completion		
Soil, sediment and groundwater sampling	Tetra Tech	May 2013	June 2013	Category 1 - Draft DGA Report	December 8, 2013

SAP Worksheet #17 -- Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)

The sampling design for this project is based on the need to fill data gaps that exist after the completion of various other site investigations and remedial actions conducted by the DESC. To fill the data gaps, Tetra Tech has expanded the list of analytes and the sampling areas in places to be investigated under this SAP. The sampling SOPs for groundwater and soil sampling for both categories are identified in Worksheet #18 and included in Appendix B. Project-specific sampling procedures are detailed in Appendix D.

AOC 001

AOC 001 was originally screened and tested for a limited number of analytes in soil, mainly TPH, using Petroflag™ screening and/or laboratory analyses. The sand filter/burn chamber was reported to have previously been used for burning of tank sludge. Given the site history, the Project Team decided to expand the analyte list to include contaminants such as metals that may be present due to burning sludge and expand the sampling area and the number of soil samples being collected in these areas. One groundwater sample (from GZ-301) will also be collected in this area to confirm previous results indicating no impact to groundwater.

The soil sampling for this project is designed to re-sample areas of highest contamination previously detected on the west and south sides of the former burn chamber and to step out approximately 10 feet from those locations in an attempt to delineate soil contamination in the lateral direction. In addition, locations adjacent to the former sampling stations on the north and east side of the former burn chamber were also selected for sampling. Two sampling locations along the discharge line from the burn chamber were also selected, but could be adjusted pending the results of the geophysical investigation (Worksheet #14). Locations of potential releases from the piping will be targeted. At each location two samples will be collected, one surface soil sample and one subsurface soil sample, in an attempt to delineate contamination vertically.

The sampling area for AOC 001 is focusing on areas of potentially contaminated soil, based upon previous data and historical record. This sampling technique makes the results of the analyses conservative in that they likely represent worst-case scenario soil conditions within the AOC. Sampling sites (Figure 3) will be located in the field using a global positioning system (GPS). Field geophysical methods to determine the location of the discharge pipe (Worksheet #14). The locations of the previous and proposed samples that will be collected are shown on Figure 3.

Eight soil borings will be advanced using direct push technology (DPT) or conventional drilling methods at the discretion of the Project Manager who will base this decision on field conditions. The burn chamber

structure was reportedly constructed on top of bedrock (Tetra Tech EC, 2005). Therefore, borings will be advanced to the top of bedrock. Surface soil samples will be collected from the 0 to 1 foot interval. The subsurface soil sample will be selected collected based on PID headspace screening and visual and olfactory observations, in order to collect the subsurface sample from the most contaminated depth, based on the discretion of the FOL. Project specific field procedures are described in Appendix D. Soil samples will be analyzed for VOCs, TPH (GRO and ExTPH), PAHs, metals, and dioxins. The number of sampling locations has been summarized in Table 17-1. The locations where field duplicate and QC samples are collected will be determined in the field by the FOL. Field duplicates will be collected where detectable levels of contamination are expected to be found based on field conditions and screening measurements.

Sediment samples will also be collected from the AOC 001 discharge area in the wetland to the immediate north and within Lawton's Brook. Six locations are presented on Figure 3 (subject to change during the site walk depending on conditions). All locations will be sampled at the 0-6 inch interval. A one-foot interval sediment sample will also be collected from SD03, SD04, and, SD05 at a depth of between 6 inches and 5 feet. The one-foot interval will be chosen in the field, determined by visual or olfactory evidence, or fine grained material if visual or olfactory evidence is not observed. Shallow sediment samples will be analyzed for VOCs, TPH (GRO and ExTPH), PAHs, metals and dioxins. Deeper sediment samples will be analyzed for VOCs, TPH (GRO and ExTPH), PAHs and metals.

The water body at the Site is a wetland and is a low energy environment. A storm event that would cause scouring of the wetland and expose deeper sediment is unlikely. Therefore, the deeper sediment samples are going to be analyzed for nature and extent purposes and not risk assessment purposes.

Building 227

This building contains an electrical transformer and batteries. With the exception of an investigation for TPH in soil around a presumed pipe on the south side of the building, and the sampling of groundwater downgradient of the structure (monitoring well GZ-318), investigation of this building has been limited. Soil around this presumed pipe was originally screened for TPH using Petroflag™ screening. Given the historical use of this building, the Project Team decided to expand the analyte list for soil to include contaminants (PCBs, metals, ExTPH and GRO) that may be present due to the use of this structure as an ECH. Additional groundwater samples will be collected from existing monitoring well TF3-ECH-GZ-318 and newly installed monitoring well TF3-ECH-MW01to confirm previous results indicating no impact to groundwater using an expanded target analyte list.

The targeted sampling areas are each side of the building and the pipe on the south side of the building. The sampling locations (Figure 4) around the building are approximate and may be adjusted in the field to collect samples adjacent to all doors to the building.

Eight soil borings will be advanced using DPT or conventional drilling methods at the discretion of the Project Manager who will base this decision on field conditions. Borings will be advanced to 10 feet bgs. Soil samples will be collected from the 0 to 1 foot interval. The subsurface soil samples around the building will be collected from the 2 to 4 foot interval, which, based on the presumption of a release to surface soil, would be the most contaminated subsurface soil interval. In addition, subsurface soil depths around the presumed pipe will be collected from the depth directly beneath the pipe. All soil samples will be analyzed for PCBs and metals. The number of sampling locations has been summarized in Table 17-1. The locations where field duplicate and QC samples are collected will be determined in the field by the FOL. Field duplicates will be collected where detectable levels of contamination are expected to be found, based on field conditions and screening measurements.

AOC 020

This AOC currently contains two pad mounted transformers around which PCBs have been detected in surface soils. PCBs were detected on the north side of transformer 1 (TF3-TF1-D) and on the east (TF3-TF2-C) and west (TF3-TF2-A) sides of transformer 2. Soil sampling is designed to confirm the PCBs in the three locations and to step out from each location in two horizontal directions and in the vertical (Figure 5). New soil boring locations will be 10 to 15 feet from the old locations with detections of PCBs. The sampling program has also been designed to determine if PCBs are present in subsurface soils, by collecting subsurface soil samples from each location. Figure 5 shows the locations of the previous soil samples and the locations of the proposed soil samples.

Eight soil borings will be advanced using DPT or conventional drilling methods at the discretion of the Project Manager. Borings will be advanced to 10 feet bgs. Soil samples will be collected from the 0 to 1 foot interval. Subsurface soil will be collected from 2 to 4-feet bgs, unless field observations (visual or olfactory evidence) suggest a different interval would contain more transformer oil or related contaminants. The number of sampling locations has been summarized in Table 17-1. The locations where field duplicate and QC samples are collected will be determined in the field by the FOL. Field duplicates will be collected where detectable levels of contamination are expected to be found based on field conditions and screening measurements.

One groundwater sample (from GZ-314) will also be collected in this area to determine if the PCBs in soil have impacted groundwater. Figure 5 shows the location of GZ-314.

Table 17-1 – Number of Sample Locations by Analytical Group, Matrix, Tank Farm 3 Category 1 Areas, and Depth (Soils)

Area Depth (Soils)	VOCs[1]	EDB[1]	TPH	PAHs	Metals	PCBs	Dioxins
Soil							
AOC TF3-001							
0-1' bgs	8		8	8	8		8
2' to the top of bedrock (2' interval To Be Determined [TBD])*	8		8	8	8		8
AOC TF3-020							
0-1' bgs						8	
2-10' bgs (2' interval TBD)*						8	
ECH (Building 227)							
0-1' bgs			8		8	8	
2-10' bgs (2' interval TBD)*			8		8	8	
Total Soils	**16**		**32**	**16**	**32**	**32**	**16**
Groundwater							
TF3-001-GZ301	1	1		1	1		
TF3-020-GZ314						1	
TF3-ECH-GZ318					1	1	
TF3-ECH-MW01					1	1	
Total Groundwater	**1**	**1**		**1**	**3**	**3**	
Sediment							
TF3-001-SD01 to SD06, 0-6 inches	6		6	6	6		6
TF3-001-SD03 to SD05, subsurface sample depth TBD in the field	3		3	3	3		3
Total Sediment	**9**		**9**	**9**	**9**		**9**

* - Subsurface soil sampling depths will be determined by the field geologist using the criteria described in Worksheet #17.

1. EDB is a VOC but is listed separately from VOCs for groundwater (and associated rinsate blanks) because it will be analyzed by SW-846 8011 instead of SW-846 8260B. EDB in soil and sediment (and associated rinsate blanks) will be analyzed by 8260B and is included within the VOC analytical group.

SAP Worksheet #18 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Area	Location Identification	Matrix	Depth (bgs)	Analytical Group	Number of Samples[1]	Sampling SOP Reference[2]
AOC 001	TF3-001-SB101 through TF3-001-SB108	Soil	0-1 foot	VOCs, GRO ExTPH, PAHs, Metals, Dioxins	8	GH-1.3, SA-1.3
			2-10 feet (2' interval TBD)	VOCs, GRO, ExTPH, PAHs, Metals, Dioxins	8	
	TF3-001-SD01 through TF3-001-SD06	Sediment	0-0.5 feet	VOCs, GRO, ExTPH, PAHs, Metals, Dioxins	6	SA-1.2
	TF3-001-SD03 through TF3-001-SD05	Sediment	0.5 to 5 feet (TBD)	VOCs, GRO ExTPH, PAHs, Metals	3	SA-1.2
	GZ301	Groundwater	Varies	VOC, EDB[3], PAHs, Metals	1	GW001 (EPA)
AOC 020	TF3-020-SB101 through TF3-020-SB108	Soil	0-1 foot	PCBs	8	GH-1.3, SA-1.3
			2-10 feet (2' interval TBD)	PCBs	8	
	GZ314	Groundwater	Varies	PCBs	1	GW001 (EPA)
Building 227	TF3-ECH-SB101 through TF3-ECH-SB108	Soil	0-1 foot	PCBs, GRO, ExTPH, Metals	8	GH-1.3, SA-1.3
			2-10 feet (2' interval TBD)	PCBs, TPH, Metals	8	
	GZ318 and ECH-MW01	Groundwater	Varies	PCBs, Metals	2	GH-1.3, GW001 (EPA)

Notes:
1. Field duplicates will be selected based on field conditions at the time of the sampling event and are not included on this worksheet, QC samples are listed on WS#20.
2. Refer to Worksheet #21 for complete reference. SOPs are included in Appendix B. Project-specific sampling procedures are provided in Appendix D.
3. For groundwater, EDB is listed separately from VOCs because EDB will be analyzed by a different method. (EDB in soil and sediment will be analyzed within the VOC analytical group.)

SAP Worksheet #19 -- Analytical SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Containers[2] (number, size, and type)	Sample volume[3] (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[4] (preparation / analysis)
Soil and Sediment	VOCs	SW-846 5035A, 8260B/CA-202, CA-214	Two 40-milliliter (mL) VOC vials	5 grams (g)	1 g sodium bisulfate in 5 ml reagent water; cool to ≤ 6 °C	14 days to analysis
			One 40-mL VOC vial	5 g	5 ml methanol, cool to ≤ 6 °C	
			One 2-ounce (oz) jar for % moisture	5 g	Cool to ≤ 6 °C	
	GRO	SW-846 5035A, 8015C/ CA-316	Two 40-mL VOC vials	5 g	5 ml methanol, cool to ≤ 6 oC	14 days to analysis
	ExTPH	SW-846 3540C or 3550C, 8015C/ CA-315, CA-527, CA-535	8-oz wide mouth jar	30 g	Cool to ≤ 6 °C	14 days to extraction; 40 days to analysis
	PAHs	SW-846 3540C or 3550C, 8270D SIM/CA-213, CA-512, CA-526		30 g	Cool to ≤ 6 °C	14 days to extraction; 40 days to analysis
	PCBs (soil only)	SW-846 3540C, 3545A or 3550C, 8082A/CA-329, CA-500, CA-524, CA-537		30 g	Cool to ≤ 6 °C	30 days to extraction; 40 days from extraction to analysis[5]
	Metals	SW-846 3050B, 6020A, 7471B/ CA-605, CA-611, CA-627	4-oz wide mouth jar	2 g	Mercury: Cool to ≤ 6 °C	180 days to analysis except for mercury; 28 days to analysis for mercury
	Dioxins	SW-846 8290/ WS-IDP-0005, WS-ID-0005	One 8-ounce glass jar with Teflon®-lined lid or stainless steel liner	30 grams	Cool to ≤ 6 °C	30 days to extraction; 45 days to analysis

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Containers[2] (number, size, and type)	Sample volume[3] (units)	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[4] (preparation / analysis)
Aqueous (Groundwater and Rinsate Blanks)	VOCs	SW-846 5030B, 8260B/ CA-202	Two 40- mL VOC vials	40 mL	Hydrochloric acid (HCl) to pH < 2, cool to ≤ 6 °C	14 days to analysis
	EDB[6]	SW-846 8011/ CA-391	Two 40-mL VOC vials	40 mL	HCl to pH < 2, no headspace, cool to ≤ 6 °C	14 days to analysis
	GRO	SW-846 5030B, 8015C / CA-316	Two 40-mL VOC vials	40 mL	HCl to pH < 2, no headspace cool to ≤ 6 oC	14 days to analysis
	PAHs	SW-846 3510C or 3520C, 8270D SIM/ CA-213, CA-502	Two 1-liter (L) amber glass bottles	1000 mL	Cool to ≤ 6 °C	7 days to extraction; 40 days to analysis
	ExTPH	SW-846 3510C or 3520C, 8015C/ CA-315, CA-520	Two 1-L amber glass bottles	1000 mL	Cool to ≤ 6 oC	7 days to extraction; 40 days to analysis
	PCBs	SW-846 3510C or 3520C, 8082A/ CA-329, CA-515	Two 1-L amber glass bottles	1000 mL	Cool to ≤ 6 °C	30 days to extraction; 40 days from extraction to analysis[5]
	Metals	SW-846 3010A, 6020A, 7470A/ CA-604, CA-615, CA-627	500 mL polyethylene bottle	100 mL	Nitric acid to pH < 2	180 days to analysis except for mercury; 28 days to analysis for mercury
	Dioxins (rinsate blanks only)	SW-846 8290/ WS-IDP-0005, WS-ID-0005	Two 1-L Amber Glass Bottles	1 L	Cool to ≤ 6 °C	30 days to extraction; 45 days to analysis

Notes:
1. Refer to the Analytical SOP References table (Worksheet #23).
2. Laboratories may provide specific containers at their discretion.
3. Minimum sample volume or mass requirement.
4. Maximum holding time is calculated from the time the sample is collected to the time the sample is extracted, digested, or analyzed.
5. SW-846 8082A does not specify a holding time to extraction; 30 days is presented here as a conservative limit. The method recommends a holding time of 40 days from extraction to analysis for extracts stored under refrigeration in the dark; but it also refers to SW-846 Chapter 4, which specifies that there is no holding time for PCBs. Additionally, SW-846 8082A states that the holding times listed in the method under the conditions listed (apparently referring to storage of extracts) may be as long as a year.
6. EDB analysis by SW846 8011 is only required for groundwater and associated rinsate blanks. Rinsate blanks associated with soil and sediment samples will be analyzed for VOCs by SW-846 8260B, for the same target VOCs (including EDB) as are required for soil and sediment in Worksheets #15a and #15c.

SAP Worksheet #20 -- Field Quality Control Sample Summary Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates[1]	No. of Assigned Laboratory QC Samples[2]	No. of Field Blanks	No. of Equip. Blanks[3]	No. of VOA Trip Blanks[4]	No. of PT Samples	Total No. of Samples to Lab[5]
Soil	VOCs	16	2	1	0	1	4	0	23
	PAHs	16	2	1	0	1	0	0	19
	PCBs	32	4	2	0	2	0	0	38
	Metals	32	4	2	0	2	0	0	38
	Dioxins	16	2	1	0	1	0	0	19
	ExTPH	32	3	2	0	2	0	0	37
	GRO	32	3	2	0	2	4	0	41
Groundwater	VOCs	1	1	1	0	1	1	0	4
	EDB	1	1	1	0	1	1	0	4
	PAHs	1	1	1	0	1	0	0	3
	PCBs	3	1	1	0	1	0	0	5
	Metals	3	1	1	0	1	0	0	5
Sediment	VOCs	9	1	1	0	1	1	0	12
	PAHs	9	1	1	0	1	0	0	11
	Metals	9	1	1	0	1	0	0	11
	Dioxins	6	1	1	0	1	0	0	8
	ExTPH	9	1	1	0	1	0	0	11
	GRO	9	1	1	0	1	0	0	11

Notes:

1. Collect 1 field duplicate per 10 field samples for each matrix.
2. Assign 1 Laboratory QC sample per 20 samples for MS/MSD analysis for organics and MS/laboratory duplicate analysis for metals. Collect triple volume of groundwater for organic analyses and double volume for metals analysis. Triple volume is also required for VOC soil and sediment. For other soil analytes, no extra volume is required. Extra volume of sediment may be needed if samples have high moisture content.
3. Collect 1 rinsate blank per 20 field samples. The rinsate blank is collected by running deionized water through any non-dedicated, non-disposable equipment after decontamination and prior to use.
4. In each cooler containing volatile samples (VOCs and EDB), ship one trip blank for each volatile analytical group. Trip blanks are pre-preserved VOC and EDB sample containers (as described in Worksheet #19) prepared by the laboratory.
5. Total number of samples does not include the assigned laboratory QC samples.

SAP Worksheet #21 -- Project Sampling SOP References Table

(UFP-QAPP Manual Section 3.1.2)

Reference Number	Title, Revision Date and / or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work?	Comments[1]
CT-05	CT-05 - Database Records and Quality Assurance; Revision 2, 2001	Tetra Tech	Not applicable	No	
GH-1.1	GH-1.1 – Site Recon; Revision 1, 1999	Tetra Tech	Camera, Tape Measure, Stakes etc.	No	
GH-1.2	GH-1.2 - Evaluation of Existing Monitoring Wells and Water Level Measurement; Revision 2, 2003	Tetra Tech	Electronic water level indicator	No	.
GH-1.3	GH-1.3 - Soil and Rock Drilling Methods; Revision 1, 1999	Tetra Tech	Drilling rig and accessories	Yes	Modifications include removal of select drilling methods
GH-1.5	GH-1.5 - Borehole and Sample Logging; Revision 1, 1999	Tetra Tech	Not applicable	No	
GH-3.1	GH-3.1 – Resistivity and Electromagnetic Induction	Tetra Tech	EM and Resistivity Equipment	No	
GH-3.2	GH-3.2 – Magnetic and Metal Detection Surveys	Tetra Tech	Metal Detection Equipment	No	
GH-3.3	GH-3.3 – Seismic Refraction Survey	Tetra Tech	Seismic Refraction Equipment	No	
GH-3.4	GH-3.4 – Ground-Penetrating Radar (GPR) Surveys	Tetra Tech	GPR Equipment	No	
HS-1.0	HS-1.0 - Utility Locating and Excavation Clearance; Revision 2, 2003	Tetra Tech	Remote subsurface sensing, magnetometer, GPR, etc.	No	
SA-1.2	SA-1.2 – Surface Water and Sediment Sampling; Revision 5, March 2008	Tetra Tech	Sampling supplies	No/Yes	Modifications include removal of surface water sampling techniques

Reference Number	Title, Revision Date and / or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work?	Comments[1]
SA-1.3	SA-1.3 - Soil Sampling; Revision 9, 2009	Tetra Tech	Sampling supplies	Yes	Also see Appendix D for project-specific procedures to augment SOP. Modifications include removal of Test Pitting Operations
SA-6.1	SA-6.1 - Non-Radiological Sample Handling; Revision 3, 2004	Tetra Tech	Not applicable	No	
SA-6.3	SA-6.3 - Field Documentation; Revision 3, 2009	Tetra Tech	Not applicable	Yes	Modifications include removal of documentation processes not being utilized for TF3 investigation
SA-7.1	SA-7.1 - Decontamination of Field Equipment; Revision 6, 2009	Tetra Tech	Not applicable	No	
GW 001	Low Stress (Low Flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells; Revision 3, 2010 / GW 001	EPA Region 1	Submersible pump	Yes (2)	Also see Appendix D for project-specific procedures to augment SOP.
	Calibration of Field Instruments; Revision 2, 2010	EPA Region 1	Multi-probe Water Quality Instrument	No	No Reference number provided

Notes:
1. SOPs are included as Appendix B. Appendix D contains project specific sampling procedures.
2. If saturated screen length is greater than 10 feet, the sampling procedures will be modified as described in Appendix D.

SAP Worksheet #22 -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Activity[1]	Frequency	Acceptance Criteria	Corrective Action	Resp. Person	SOP Reference	Comments
YSI 600 Series Water Quality Meter	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's guidance	Operator correction or Replacement	Field Operations Leader	Manufacturer's instruction manual and EPA Region 1- Calibration of Field Instruments	Rental field equipment will be used
Turbidity Meter	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's guidance	Operator correction or Replacement	Field Operations Leader	Manufacturer's instruction manual and EPA Region 1- Calibration of Field Instruments	
Water Level Indicator	Visual Inspection Field checks as per manufacturer	Daily Once on receiving from vendor	0.01 foot accuracy	Operator correction or Replacement	Field Operations Leader	Manufacturer's instruction manual	
Photo-Ionization Detector (PID)/ Flame Ionization Detector (FID)	Visual Inspection Calibration/ Verification	Daily Beginning and end of day	Manufacturer's guidance	Operator correction or Replacement	Field Operations Leader	Manufacturer's instruction manual	

[1]Rental equipment and instruments will be used in the field. The rental firms will be responsible for the proper care, maintenance, and repair of these items, and for tracking and documenting equipment and instrument maintenance and repairs.

SAP Worksheet #23 -- Analytical SOP References Table

(UFP-QAPP Manual Section 3.2.1)

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
CA-202	Analysis of VOAs by Purge and Trap GC/MS: SW-846 Method 8260, 04/10, Revision 11.	Definitive	Soil, Sediment, and Water/VOCs	Gas Chromatography (GC)/Mass Spectroscopy (MS)	Katahdin	N
CA-213	Analysis of Semivolatile Organic Compounds By: SW 846 Method 8270 – Modified For SIM, 04/10, Revision 8.	Definitive	Soil, Sediment, and Water/PAHs	GC/MS	Katahdin	N
CA-214	Closed-System Purge-And-Trap And Extraction For Volatile Organics In Soil And Waste Samples Using SW846 Method 5035, 09/08, Revision 5.	Definitive	Soil and Sediment/VOCs	Not applicable (extraction)	Katahdin	N
CA-315	Determination of Extractable Petroleum Hydrocarbons or DRO by Modified Methods 8015 and 8100, 04/10, Revision 10.	Definitive	Water and Soil/ ExTPH	GC/FID	Katahdin	N
CA-316	Method for Determining Volatile Petroleum Hydrocarbons or GRO by Modified Method 8015, 08/09, Revision 9.	Definitive	Water and Soil/ GRO	GC/FID	Katahdin	N
CA-319	Extraction and Analysis of EDB (1,2-Dibromoethane) and DBCP (1,2-Dibromo-3-chloropropane) in Water by SW846 Method 8011, 12/10, Revision 7.	Definitive	Water/EDB	GC/Electron Capture Detector (ECD)	Katahdin	N
CA-329	Analysis Of PCBs As Total Aroclors By GC/ECD: SW-846 Method 8082, 04/10, Revision 11.	Definitive	Soil and Water/PCBs	GC/ECD	Katahdin	N

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
CA-500	Preparation Of Sediment/Soil Samples By Sonication Using Method 3550 For Subsequent Pesticides/PCBs Analysis, 08/10, Revision 7.	Definitive	Soil/PCBs	Not applicable (extraction)	Katahdin	N
CA-502	Preparation Of Aqueous Samples For Extractable Semivolatile Analysis, 10/09, Revision 6.	Definitive	Water/PAHs	Not applicable (extraction)	Katahdin	N
CA-512	Preparation Of Sediment/Soil Samples By Sonication Using Method 3550 For Subsequent Extractable Semi-Volatiles Analysis, 08/10, Revision 8.	Definitive	Soil and Sediment/ PAHs	Not applicable (extraction)	Katahdin	N
CA-515	Preparation of Aqueous Samples for Pesticides/PCBs Analysis, 08/10, Revision 7.	Definitive	Water/PCBs	Not applicable (extraction)	Katahdin	N
CA-524	Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For Pesticide/PCB Analysis, 08/10, Revision 7.	Definitive	Soil/PCBs	Not applicable (extraction)	Katahdin	N
CA-526	Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For Subsequent Extractable Semivolatile Analysis, 08/10, Revision 7.	Definitive	Soil and Sediment/ PAHs	Not applicable (extraction)	Katahdin	N
CA-527	Preparation Of Sediment/Soil Samples By Soxhlet Extraction Using Method 3540 For Subsequent ExTPH or DRO Analysis, 09/10, Revision 6.	Definitive	Soil/ExTPH Extraction	Not applicable (extraction)	Katahdin	N
CA-535	Preparation of Sediment/Soil Samples By Sonication Using Method 3550 For Subsequent DRO or TPH Analysis, 08/10, Revision 7.	Definitive	Soil/ExTPH Extraction	Not applicable (extraction)	Katahdin	N

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
CA-537	Preparation of Sediment/Soil and Tissue Samples by Accelerated Solvent Extraction Using Method 3545 for Subsequent Extractable Pesticide and PCB Analysis, 12/10, Revision 3.	Definitive	Soil/PCBs	Not applicable (extraction)	Katahdin	N
CA-604	Acid Digestion of Aqueous Samples by EPA Method 3010 for ICP and ICP-MS Analysis of Total or Dissolved Metals, 04/10, Revision 5.	Definitive	Water/Metals	Not applicable (digestion)	Katahdin	N
CA-605	Acid Digestion of Solid Samples by USEPA Method 3050 for Metals by ICP-AES and GFAA, 09/10, Revision 5.	Definitive	Soil and Sediment/Metals	Not applicable (digestion)	Katahdin	N
CA-611	Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471, 12/10, Revision 8.	Definitive	Soil and Sediment/Mercury	Mercury Analyzer	Katahdin	N
CA-615	Digestion and Analysis of Aqueous Samples for Mercury by USEPA Method 7470, 04/10, Revision 5.	Definitive	Water/Mercury	Mercury Analyzer	Katahdin	N
CA-627	Trace Metals Analysis By ICP-MS Using USEPA Method 6020, 04/10, Revision 7.	Definitive	Soil, Sediment, and Water/Metals	ICP-MS	Katahdin	N
WS-ID-0005	Analysis of Samples for Polychlorinated Dioxins and Furans by HRGC/HRMS 12/09, Revision 7.3.	Definitive	Soil, Sediment, and Water/ Dioxins	GC/HRMS	TestAmerica West Sacramento	N
WS-IDP-0005	Preparation of Samples for Analysis of Polychlorinated Dioxins and Furans for Analysis HRGC/HRMS, 02/10, Revision 1.1.	Definitive	Soil, Sediment, and Water/ Dioxins Extraction	Not applicable (extraction)	TestAmerica West Sacramento	N

SAP Worksheet #24 -- Analytical Instrument Calibration Table

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
GC/MS VOCs	Initial Calibration (ICAL) - A minimum 5-point calibration is required.	Calibrate the instrument when it is received and after a major change or if the daily calibration fails.	The average Response Factors (RFs) for System Performance Check Compound (SPCCs) must be ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorobenzene and ≥ 0.10 for chloromethane, 1,1-dichloroethane and bromoform. The Percent Relative Standard Deviation (%RSD) for RFs for Calibration Check Compounds (CCCs) must be ≤ 30%, and one option below must be met: Option 1) %RSD < 15% for all other compounds. If not met: Option 2) Linear least squares regression: correlation coefficient (r) ≥ 0.995. Option 3) Non-linear regression: coefficient of determination (r^2) ≥ 0.99 (6 points for second order).	Repeat calibration if criterion is not met	Analyst, Department Manager	CA-202
	Initial Calibration Verification (ICV) (Second Source)	Once after each ICAL.	The Percent Recovery (%R) must be within 80-120% for all target compounds.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	
	Establish Retention Time (RT) Window Position	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.		
	Evaluation of Relative Retention Times (RRTs)	With each sample.	RRT of each target compound must be within ±0.06 RPT units.	Correct problem, then rerun ICAL.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
	Continuing Calibration Verification (CCV)	Analyze a standard at the beginning of each 12-hour shift after a bromofluorobenzene (BFB) tune.	The RFs for SPCCs must be ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorobenzene and ≥ 0.10 for chloromethane, 1,1-dichloroethane and bromoform. The %D for all target compounds and surrogates must be ≤ 20% (D = Difference or Drift).	Department of Defense (DoD) project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	Analyst, Department Manager	
	BFB Tune	Every 12 hours.	Criteria listed in Section 7.3, current revision of SOPs CA-202.	Retune and/or clean source.	Analyst, Department Manager	
GC/ECD EDB[(2)]	ICAL	Instrument receipt, major instrument change, when CV does not meet criteria	One of the options below must be met: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995; Option 3: non-linear regression: coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order).	(1) Perform instrument maintenance as needed. (2) Reanalyze and or reprep calibration standards.	Analyst, Department Manager	CA-319
	ICV	Immediately following ICAL.	All project analytes must be within established retention time windows. All project analytes must be within ± 20% of expected value from the ICAL.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	
	Establish RT Window Position	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	Not applicable.	Analyst, Department Manager	
	CCV	Daily prior to sample analysis and at intervals of not less than once every 20 samples. Also, at the end of the analysis sequence.	%D must be ≤ +/- 20%.	(1) Evaluate the samples: If the %D>±20% and sample results are <PQL, narrate. If %D>±20% and is likely a result of matrix interference, narrate. All samples must be reanalyzed that fall within the standard that exceeded criteria and the last standard that was acceptable.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
GC/MS (SIM) PAHs	Decafluorotriphenyl-phosphine (DFTPP) Tune.	Beginning of each analytical run or every 12 hours	DFTPP within method specifications for tuning criteria.	Re-tune instrument, clean MS source as needed.	Analyst, Department Manager	CA-213
	ICAL - 5-7 (minimum 5 points required) calibration standards, initial calibration for all analytes.	Instrument receipt, major instrument change, when continuing calibration verification (CCV) does not meet criteria.	Project specific criteria: Average Response Factor (RF) for all PAHs must be ≥ 0.05. Percent Relative Standard Deviation (%RSD) for RFs for all PAHs must be ≤ 30% or one option below: Option 1) Linear least squares regression: correlation coefficient (r) must be ≥ 0.995 Option 2) Non-linear regression: coefficient of determination (r^2) must be ≥ 0.99 (6 points for second order).	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst, Department Manager	
	ICV (Second Source)	Once after each ICAL.	Percent recovery (%R) must be within 80-120% for all target compounds.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	
	Establish Retention Time (RT) Window Position	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	Not applicable.	Analyst, Department Manager	
GCMS – PAHs (cont.)	Evaluation of Relative Retention Times (RRTs)	With each sample.	RRT of each target compound must be within ±0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
	CCV	Daily before sample analysis and every 12 hours	Project specific criteria: For all PAHs RF must be ≥ 0.05 All PAHs and surrogates must be ≤ 25%D (D = Difference or Drift);	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	Analyst, Department Manager	
GC/ECD PCBs	ICAL - A minimum 5-point calibration is required.	Instrument receipt, major instrument change, when CCV does not meet criteria.	6 point calibration of Aroclors 1016/1260, 1242, 1248, and 1254 – One of the options below must be met: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995; Option 3: non-linear regression: r^2 ≥ 0.99 (6 points shall be used for second order) Mid-point calibration of Aroclors 1221 and 1232; if targets are detected, 6-point calibration is performed.	Repeat ICAL and/or perform necessary equipment maintenance. Check calibration standards. Reanalyze affected data.	Analyst, Department Manager	CA-329
	ICV	Aroclors 1016 and 1260: Once after each ICAL.	The %D must be ≦ 20%D for Aroclors 1016 and 1260. (D = Difference or Drift)	Correct problem, rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	
	CCV	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	The %D must be ≦ 20%D for all project analytes.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	
ICP-MS	Tune	Daily prior to calibration	Mass calibration must be within 0.1 amu of true value, Resolution must be < 0.9 amu at 10% peak height. RSD must be ≤ 5% for at least four replicate analyses.	Perform necessary equipment maintenance.	Analyst, Department Manager	CA-627

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
	ICAL	Daily prior to sample analysis.	4 point calibration plus blank – The r must be ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	
	ICV (Second Source)	Once after each ICAL, and before beginning a sample run.	The %R must be within 90-110% for all analytes.	Do not use results for failing elements unless the ICV > 110% and the sample results are non-detect. Investigate and correct problem.	Analyst, Department Manager	
	Calibration Blank	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence.	No analytes detected > LOD. For negative blanks, absolute value < LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst, Department Manager	
ICP-MS (cont.)	CCV	After every 10 samples and at the end of each run sequence.	The %R must be within 90-110% for all analytes.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	
	Low-level Calibration Check Standard	Daily, after one-point ICAL.	The %R must be within 80-120% for all analytes.	Do not use results for failing elements, unless low-level standard recovery > upper limit and sample results are non-detect. Investigate and correct the problem.	Analyst, Department Manager	
	Interference Check Sample (ICS) - ICSA & ICSB	Daily, before sample injections	The absolute value of the ICSA concentration for all non-spiked analytes (except verified trace impurities) must be less than the LOD (3), and ICSB %Rs must be within 80-120%.	Correct the problem, then re-prepare checks and reanalyze all affected samples.	Analyst / Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
Mercury analyzer	ICAL	On instrument receipt, major instrument change, at the start of each day.	Initial Calibration, 5 points plus a calibration blank - r ≥ 0.995.	Recalibrate and/or perform necessary equipment maintenance. Check calibration standards.	Analyst, Department Manager	CA-611, CA-615
	ICV (Second Source)	Once after each ICAL, prior to beginning a sample run.	The %R must be within 90-110% for mercury.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Analyst, Department Manager	
	Calibration Blank	Before beginning a sample sequence, after every 10 samples and at end of the analysis sequence.	No analytes detected > LOD. For negative blanks, absolute value must be < LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Analyst, Department Manager	
	CCV	Beginning and end of each run sequence and every 10 samples.	The %R must be within 80-120% for mercury.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	Analyst, Department Manager	
GC/HRMS	Tune / Mass Resolution Check (PFK)	At the beginning and the end of each 12-hour period of analysis.	Resolving power ≥ 10,000 at m/z=304.9842 & m/z=380.9760 + 5ppm of expected mass. Lock-mass ion between lowest and highest masses for each descriptor and level of reference ≤ 10% full-scale deflection.	Retune instrument & verify. Assess data for impact if end resolution is less than 10,000 narrate or reinject as necessary.	Analyst /Lab Manager	WS-ID-0005
HRGC/HRMS	GC Column Performance Check (CPSM/WDM per method)	Prior to ICAL or calibration verification.	Peak separation between 2,3,7,8-TCDD and other TCDD isomers result in a valley of ≤ 25%; and identification of all first and last eluters of the eight homologue retention time windows and documentation by labeling (F/L) on the chromatogram; and absolute retention times for switching from one homologous series to the next ≥ 10 seconds for all components of the mixture.	1) Readjust windows. 2) Evaluate system. 3) Perform maintenance. 4) Reanalyze CPSM. 5) No corrective action is necessary if 2,3,7,8-TCDD is not detected and the % valley is greater than 25%.		

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
GC/HRMS	ICAL = Minimum five-point initial calibration for target analytes, lowest concentration standard at or near the reporting limit.	ICAL prior to sample analysis, as needed by the failure of calibration verification, and when a new lot is used as a standard source for calibration verification, internal standard or recovery standard solutions.	RSD ≤ 20% for response factors for 17 unlabeled isomers & 9 labeled IS, and ion abundance ratios within limits specified in SOP; and S/N ≥ 10:1for target analytes.	Evaluate standard and instrument response. If problem with instrument (autosampler failure, response poor, etc.) or standards, correct as appropriate, then repeat initial calibration.		
GC/HRMS	ICV	Immediately following ICAL.	All project analytes must be within ± 30% of the expected value from the ICAL.	Evaluate standards and instrument response. If standard issue, repeat or remake then repeat standard as appropriate. If still fails, repeat initial calibration	Lab Manager / Analyst	WS-ID-0005

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
GC/HRMS	CCV	At the beginning of each 12-hour period, and at the end of each analytical sequence.	Ion abundance ratios must be in accordance with SOP; and RF (unlabeled standards) within ± 20%D of average RF from ICAL; and RF (labeled standards) within ± 30%D of average RF from ICAL.	Correct problem, repeat calibration verification. If fails, repeat ICAL and reanalyze all samples analyzed since last successful CCV End of Run CCV: If RF (unlabeled standards) > ± 20%D and ≤ ± 25%D and/or RF (labeled standards) > ± 30%D and ≤ ± 35%D of the average RF from ICAL use mean RF from bracketing CCVs to quantitate impacted samples. If bracketing CCVs differ by more than 25% RPD (unlabeled) or 35% RPD (labeled), run a new ICAL within 2 hours, and requantitate samples. Otherwise, reanalyze samples with positive detections.	Lab Manager / Analyst	WS-ID-0005
GC/FID ExTPH	ICAL	Instrument receipt, major instrument change, when CV does not meet criteria	One of the options below must be met: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995; Option 3: non-linear regression: COD r2 ≥ 0.99 (6 points shall be used for second order).	(1) Perform instrument maintenance as needed. (2) Reanalyze and or reprep calibration standards.	Analyst, Department Manager	CA-315
	ICV	Immediately following ICAL.	All project analytes must be within established retention time windows. All project analytes must be within ± 20% of expected value from the ICAL.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
	CCV	Daily prior to sample analysis and at intervals of not less than once every 20 samples. Also, at the end of the analysis sequence.	%D must be ≤ +/- 20%	(1) Evaluate the samples: If the %D>+20% and sample results are <PQL, narrate. If %D>±20% and is likely a result of matrix interference, narrate. All samples must be reanalyzed that fall within the standard that exceeded criteria and the last standard that was acceptable.	Analyst, Department Manager	
GC/FID GRO	ICAL	Minimum 5 point calibration using a gasoline component mixture.	One of the options below must be met: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995; Option 3: non-linear regression: COD r2 ≥ 0.99 (6 points shall be used for second order).	Repeat initial calibration.	Analyst, Department Manager	CA-316
	ICV	Immediately following ICAL.	All project analytes within established retention time windows. All project analytes within ± 20% of expected value from the ICAL.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Analyst, Department Manager	
	CCV	Daily prior to sample analysis and at intervals of not less than once every 20 samples. Also, at the end of the analysis sequence.	%D must be ≤ +/- 20%.	Evaluate the samples – if the %D>+20% and sample results are <PQL, narrate. If %D> □2 result of matrix interference, narrate. All samples must be reanalyzed that fall within the standard that exceeded criteria and the last standard that was acceptable	Analyst, Department Manager	

Notes:
1. Refer to the Analytical SOP References table (Worksheet #23).
2. EDB analysis by GC/ECD is only applicable for analysis of groundwater and associated rinsate blank samples. EDB analysis in soil and sediment will be by GC/MS, as part of the VOC analytical group.
3. For data validation, the criterion for ICSA will be < LOQ.

SAP Worksheet #25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.2.3)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person[2]	SOP Reference
GC/MS	Check pressure and gas supply daily. Bake out trap and column, manual tune if BFB not in criteria, change septa as needed, cut column as needed, change trap as needed. Other maintenance specified in lab Equipment Maintenance SOP.	VOCs	Ion source, injector liner, column, column flow, purge lines, purge flow, trap.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	CA-202
GC/MS	Check pressure and gas supply daily. Manual tune if DFTPP not in criteria, change septa as needed, change liner as needed, cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP	PAHs	Ion source, injector liner, column, column flow	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	CA-213
GC/ECD	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	PCBs and EDB	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	CA-329, CA-319
ICP-MS	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, replace peristaltic pump tubing as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Metals	Torch, nebulizer, spray chamber, pump tubing	Prior to ICAL and as necessary	Acceptable ICAL or CCV	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	CA-627

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	CA	Responsible Person[2]	SOP Reference
Mercury Analyzer	Replace peristaltic pump tubing, replace mercury lamp, replace drying tube, clean optical cell and/or clean liquid/gas separator as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Mercury	Tubing, sample probe, optical cell	Prior to ICAL and as necessary	Acceptable ICAL or CCV	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	CA-611, CA-615
GC/HRMS	Parameter Setup	Dioxins/ Physical check	Physical check	Initially; prior to DCC	Correct Parameters	Reset if incorrect	TestAmerica Chemist	WS-ID-0005
GC/HRMS	Tune Check	Dioxins/ Instrument Performance	Conformance to instrument tuning.	Initially; prior to DCC	Compliance to ion abundance criteria	Correct the problem and repeat tune check	TestAmerica Chemist	WS-ID-0005
GC/FID	Check pressure and gas supply daily. Change septa and/or GC injector glass liner as needed. Replace or cut GC column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	ExTPH	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	CA-315
GC/FID	Change septa, and/or GC injector glass liner as needed. Replace or cut GC column as needed. Bake out trap and column, change trap as needed.	GRO	Injector liner, septa, column, column flow.	Prior to ICAL and/or as necessary.	Acceptable ICAL or CCV	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	CA-316

SAP Worksheet #26a – Sample Handling System

(UFP-QAPP Manual Appendix A)

Sample Handling System - Katahdin

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): FOL, Tetra Tech
Sample Packaging (Personnel/Organization): FOL, Tetra Tech
Coordination of Shipment (Personnel/Organization): FOL, Tetra Tech.
Type of Shipment/Carrier: Hand carrier or overnight courier service (Federal Express)
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt: Sample Custodians / Katahdin
Sample Custody and Storage : Sample Custodians / Katahdin
Sample Preparation : Extraction Lab, Metals Preparation Lab / Katahdin
Sample Determinative Analysis : Gas Chromatography Lab, Gas Chromatography/Mass Spectrometry Lab, Metals Lab / Katahdin
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 60 days from receipt
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 3 months from sample digestion/extraction
Biological Sample Storage (No. of days from sample collection): N/A
SAMPLE DISPOSAL
Personnel/Organization: Sample Custodians/ Katahdin

SAP Worksheet #26b – Sample Handling System

(UFP-QAPP Manual Appendix A)

Sample Handling System – Test America

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Field Operation Leader, Tetra Tech
Sample Packaging (Personnel/Organization): Field Operation Leader, Tetra Tech
Coordination of Shipment (Personnel/Organization): Field Operation Leader, Tetra Tech
Type of Shipment/Carrier: Overnight courier service (FedEx)
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Custodians, TestAmerica
Sample Custody and Storage (Personnel/Organization): Sample Custodians, TestAmerica
Sample Preparation (Personnel/Organization): Sample Preparation Technicians, TestAmerica
Sample Determinative Analysis (Personnel/Organization): Sample Analysis Technicians, TestAmerica
SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 60 days
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 60 days
Biological Sample Storage (No. of days from sample collection): Not applicable
SAMPLE DISPOSAL (Intact leftover samples)
Personnel/Organization: Sample Custodians, TestAmerica

SAP Worksheet #27 – Sample Custody Requirements Table

(UFP-QAPP Manual Section 3.3.3)

Sample Designation and Tracking System

Each sample collected will be assigned a unique sample tracking number used to catalog the results. The sample location IDs are listed in Worksheet #18. The sample tracking number for monitoring well and soil samples will consist of alpha-numeric characters identifying the site, area, sample medium, location, and depth or date. Any other pertinent information regarding sample identification will be recorded on the sample log sheets, chain-of-custody forms, or in the field logbooks.

The alpha-numeric (A-N) coding to be used in the sample system is detailed below and in the subsequent definitions.

AAA	-	NNN	-	AA-NNNN	-	NNNN
(Site ID)	-	(AOC ID)	-	(Medium & Location)	-	(Depth or Date)

Site identifier: "TF3" for Tank Farm 3

AOC identifier: AOCs investigated by TtEC during the SIRAR that are already designated with an AOC ID will utilize the same ID (AOC-001 or AOC-020). Building 227 was never designated as a separate AOC during the SI, so for this field effort samples that are collected from this area will be designated with the letters ECH.

Medium identifier:

- "SB" used to designate samples collected from soil borings
- "SD" used to designate samples collected from sediment sample locations
- "GW" used to designate groundwater samples collected from monitoring wells

Location identifier: Each sample station is assigned a unique location identifier composed of sequential numeric characters as shown on Worksheet #18. Groundwater monitoring wells will be designated as they were during the SIRAR.

Depth/Date:

For soil sample locations, this portion of the sample tracking number will represent the depth in feet bgs from which the sample was collected; e.g., for soil samples collected from 2 to 4 feet bgs, this portion of the sample tracking number will be "0204".

For sediment sample locations, this portion of the sample tracking number will represent the depth in inches bgs; e.g., a sediment sample collected from the 0-6 inch interval would be "0006".

For groundwater sample locations, this portion of the sample tracking number will represent the date the sample was collected (MMYY); e.g., a ground water sample collected in December of 2012 would be represented with "1212".

For Example: A soil sample collected from SB101 at AOC-001 from the 2-4 foot interval would be "TF3-001-SB-101-0204"; a groundwater sample collected from GZ302 on December 18, 2012 would be "TF3-001-GW-GZ302-1212".

Quality Control Samples (QC) samples collected during the investigation will use the same coding system as for the environmental samples. Field QC sample types are presented in Worksheet #20. Field QC designations will conform to the following formats:

- Field Duplicates: Blind field duplicate samples will be designated such that the location designation will be replaced with "DUP" followed by a sequential value (the nth duplicate sample collected during that sampling event) and a chronological value (MMDDYY). The sample log sheet will note from which sample location the duplicate was collected. For example, the first soil field duplicate sample collected December 1, 2012, at the Site, will be labeled TF3-SB-DUP01-120112.
- Rinsate Blanks: Rinsate blank sample identifiers will consist of the site, the medium (with "W" instead of "MW", the "RB" label, and the date (MMYY). Example: TF3-W-RB01-0811.
- Trip Blanks: will consist of the site, the medium (with "W" instead of "MW"), the "TB" label, and the date (MMYY).

Laboratory QC samples (matrix spike and laboratory duplicate samples) have no separate sample identifier codes, but are noted on the chain-of-custody record and sample log sheet.

Sample Handling and Chain-of Custody Procedures

Following collection, samples will be placed on ice in a secure cooler and attended by Tetra Tech personnel or placed in locked vehicles or designated storage areas until analysis or shipment to an off-site laboratory. Chain of custody procedures are described in further detail in the following Tetra Tech SOPs, which are provided in Appendix B:

- SA-6.3 Field Documentation.
- SA-6.1 Non-Radiological Sample Handling.

The samples will be shipped to the laboratories in coolers packed with ice and bubble wrap, or equivalent packing material, to cushion the samples to prevent breakage and to maintain the required temperature

for the samples. A container filled with water and labeled "temperature blank" will be included in each cooler. The temperature of this blank will be measured by the laboratory on sample receipt to verify acceptable sample preservation temperature. The coolers will be taped and sealed with a signed custody seal to ensure the chain of custody is maintained. The chain-of-custody forms are shipped to the laboratory with the samples.

Samples will be shipped to the laboratories by an overnight courier to ensure that maximum sample holding times are not exceeded. The maximum allowable sample holding times before sample extraction, digestion, or analysis are presented in Worksheet #19. Saturday deliveries will be coordinated by the FOL or his or her designee with the laboratory. Worksheet #19 also lists the sample containers, chemical preservatives, and temperature condition requirements to maintain the integrity of the sample. The field sampling crew will attempt to identify any potentially high concentration samples on the chain-of-custody form.

Laboratory procedures for sample receiving and chain-of-custody are detailed in SOP SD-902 (Katahdin) and SOP WS-QA-0003 (TestAmerica); and the laboratory procedures for disposal of the environmental samples are described in SOP 903 (Katahdin) and SOP WS-EHS-0001 (TestAmerica). These SOPs are included in Appendix E.

SAP Worksheet #28a – Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Note: Katahdin's statistically-derived QC limits referenced in the worksheets below refer to Katahdin's limits at the time of analysis. Katahdin's current limits are presented in Appendix E.

Matrix	Soil/Sediment/ Water
Analytical Group	VOCs
Analytical Method/ SOP Reference	SW-846 8260B / CA-202

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparation batch of twenty or fewer samples of similar matrix.	No target compounds > ½ LOQ (> LOQ for common laboratory contaminants) and > 1/10 the amount measured in any sample or 1/10 the PSL, whichever is greater.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager	Bias/Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogate	Four per sample – Dibromofluoromethane 1,2-Dichloroethane-d4 Toluene-d8 4-Bromofluorobenzene (BFB)	%R must be within Katahdin's statistically-derived QC limits.	For QC and field samples, correct problem then reprepare and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reanalyzed within hold time.	Analyst, Laboratory Department Manager	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Control Sample (LCS)	One per preparation batch of twenty or fewer samples of similar matrix.	%R must be within Katahdin's statistically-derived QC limits, allowing for the number of marginal exceedances presented in the DoD Quality Systems Manual (QSM) Version 4.1, Table G-1.	Correct problem, then reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Contact Client if samples cannot be reanalyzed within hold time.	Analyst, Laboratory Department Manager	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil/Sediment/ Water
Analytical Group	VOCs
Analytical Method/ SOP Reference	SW-846 8260B / CA-202

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
MS/MSD (not applicable for rinsate blanks)	One per sample delivery group (SDG) or every 20 samples.	%R should be within Katahdin's statistically derived QC limits. Soil/Sediment Precision: RPD should be ≤ 30%. Water Precision: RPD should be ≤ 20%.	Corrective actions will not be taken for samples when recoveries are outside limits if likely due to matrix, otherwise contact client.	Analyst, Laboratory Department Manager	Precision/Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Internal Standard (IS)	Four per sample- Pentafluorobenzene Chlorobenzene-d5 1,4-dichlorobenzene-d4 1,4-difluorobenzene	Retention times for internal standards must be ± 30 seconds and the responses within -50% to +100% of the ICAL midpoint standard.	Inspect mass spectrometer or gas chromatograph for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Laboratory Department Manager	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	Not applicable (NA)	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager	Accuracy	Same as QC Acceptance Limits.

SAP Worksheet #28b – Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Water
Analytical Group	EDB
Analytical Method/ SOP Reference	SW-846 8011 / CA-319

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparation batch of twenty or fewer samples of similar matrix.	No target compounds > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the PSL, whichever is greater.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
Surrogates	One per sample - 2,4,5,6-Tetrachloro-m-xylene	%R must be within Katahdin's statistically derived QC limits: 36-123%.	For QC and field samples, correct problem then reprepare and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepared within hold time.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparation batch of twenty or fewer samples of similar matrix.	%R must be within method limits of 60 – 140 %.	Correct problem, then reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix E). Contact Client if samples cannot be reprepared within hold time.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD (not applicable for rinsate blanks)	One per sample delivery group (SDG) or every 20 samples.	Same as LCS Water and Soil Precision: RPD should be ≤ 30%.	Corrective actions will not be taken for samples when recoveries are outside limits if likely due to matrix, otherwise contact client.	Analyst, Laboratory Department Manager, and Data Validator	Precision/Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Water
Analytical Group	EDB
Analytical Method/ SOP Reference	SW-846 8011 / CA-319

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Second Column Confirmation	All positive results must be confirmed.	Results between primary and second column must be RPD ≤ 40%. The higher of the two results will be reported unless matrix interference is apparent.	None. Apply qualifier if RPD >40% and discuss in the case narrative.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager, and Data Validator	Accuracy	Same as QC Acceptance Limits.

SAP Worksheet #28c – Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Soil/Sediment/ Water
Analytical Group	PAHs
Analytical Method/ SOP Reference	SW-846 8270D SIM / CA-213

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparation batch of twenty or fewer samples of similar matrix.	No target compounds > ½ LOQ (> LOQ for common laboratory contaminants) and > 1/10 the amount measured in any sample or 1/10 the PSL, whichever is greater.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
Surrogate	3 per sample 2-Methylnaphthalene-d10 Fluorene-d10 Pyrene-d10	%R must be within Katahdin's statistically-derived QC limits.	For QC and field samples, correct problem then reprepare and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepared within hold time.	Analyst, Laboratory Department Manager	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparation batch of twenty or fewer samples of similar matrix.	%R must be within Katahdin's statistically-derived QC limits, allowing for the number of marginal exceedances presented in DoD QSM Table G-1.	Correct problem, then reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available). Contact Client if samples cannot be reprepared within hold time.	Analyst, Laboratory Department Manager	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD (not applicable for rinsate blanks)	One per sample delivery group (SDG) or every 20 samples.	%R should be within Katahdin's statistically-derived QC limits.	Corrective actions will not be taken for samples when recoveries are outside limits if likely due to matrix, otherwise contact client.	Analyst, Laboratory Department Manager	Precision/Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil/Sediment/ Water
Analytical Group	PAHs
Analytical Method/ SOP Reference	SW-846 8270D SIM / CA-213

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
		Soil Precision: RPD should be ≤ 50%. Water Precision: RPD should be ≤ 30%.				
IS	Six per sample – 1,4-Dichlorobenzene-d4 Naphthalene-d8 Acenaphthene-d10 Phenanthrene-d10 Chrysene-d12 Perylene-d12	Retention times for internal standards must be ± 30 seconds and the responses within -50% to +100% of the ICAL midpoint.	Inspect mass spectrometer or gas chromatograph for malfunctions. Mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Laboratory Department Manager	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager	Accuracy	Same as QC Acceptance Limits.

SAP Worksheet #28d – Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Soil/Water
Analytical Group	PCBs
Analytical Method/SOP Reference	SW846 8082A /CA-329

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	Contaminants in the method blank must be < ½ LOQ,	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Re-prepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be re-prepared within hold time.	Analyst, Laboratory Department Manager and Data Validator Analyst, Laboratory Department Manager	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
Surrogates	PCBs: one per sample: Decachloro-biphenyl	%Rs must meet the laboratory statistically-derived control limits.	For QC and field samples, correct problem then re-prepare and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be re-prepared within hold time.	Analyst, Laboratory Department Manager	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparation batch of 20 or fewer samples of similar matrix.	%R must be within Katahdin's statistically-derived QC limits.	Correct problem, then re-prepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Contact Client if samples cannot be re-prepared within hold time.	Analyst, Laboratory Department Manager	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil/Water
Analytical Group	PCBs
Analytical Method/SOP Reference	SW846 8082A /CA-329

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria**
MS/MSD (not applicable for rinsate blanks)	One per SDG or every 20 samples.	%R should be within Katahdin statistically derived limits. Soil Precision: RPD should be ≤ 50%. Water Precision: RPD should be ≤ 30%.	Corrective actions will not be taken for samples when recoveries are outside limits if likely due to matrix, otherwise contact client.	Analyst, Laboratory Department Manager	Precision/Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Second Column Confirmation	All positive results must be confirmed.	Results between primary and second column must be RPD ≤ 40%. The higher of the two results will be reported unless matrix interference is apparent.	None. Apply qualifier if RPD >40% and discuss in the case narrative.	Analyst, Laboratory Department Manager	Precision	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	NA	Apply "J" qualifier to results between DL and LOQ.	NA	Analyst, Laboratory Department Manager	Accuracy	Same as QC Acceptance Limits.

SAP Worksheet #28e – Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Soil/Sediment/ Water
Analytical Group	Metals (ICP-MS)
Analytical Method/ SOP Reference	SW846 6020A / CA-627

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per digestion batch of 20 or fewer samples of similar matrix.	No target metals > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the PSL, whichever is greater. For negative blanks, absolute value < LOD.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per digestion batch of 20 or fewer samples of similar matrix.	Water and Soil: %R must be within 80-120%, allowing for the marginal exceedances presented in DoD QSM Table G-1.	Redigest and reanalyze all associated samples for affected analyte.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS (not applicable for rinsate blanks)	One per sample delivery group (SDG) or every 20 samples.	%R should be within 80-120%if sample < 4x spike added.	Flag results for affected analytes for all associated samples with ”N”.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Post-digestion Spike (not applicable for rinsate blanks)	Project-specific frequency: When MS recovery fails or analyte concentration in all samples < 50x LOD	%R should be within 75-125%.	Run associated samples by method of standard addition or flag results.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil/Sediment/ Water
Analytical Group	Metals (ICP-MS)
Analytical Method/ SOP Reference	SW846 6020A / CA-627

QC Sample:	**Frequency/ Number**	**Method/SOP QC Acceptance Limits**	**Corrective Action**	**Person(s) Responsible for Corrective Action**	**Data Quality Indicator (DQI)**	**Measurement Performance Criteria (MPC)**
Laboratory Duplicate (not applicable for rinsate blanks)	One per sample delivery group (SDG) or every 20 samples.	Project-specific criteria: If values are ≥ 5x LOQ, RPD should be ≤ 20%. If values are < 5x LOQ, Absolute Difference should be ≤ LOQ.	Flag results for affected analytes for all associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Waters: If values are ≥ 5x LOQ, RPD should be ≤ 20%; if values are < 5x LOQ, Absolute Difference should be ≤ LOQ. Soils: If values are ≥ 5x LOQ, RPD should be ≤ 35%; if values are < 5x LOQ, Absolute Difference should be ≤ 2x LOQ.
ICP Serial Dilution (not applicable for rinsate blanks)	One per preparation batch of twenty or fewer samples of similar matrix.	If original sample result is at least 50x LOQ, 5-fold dilution must agree within ± 10% of the original result.	Flag results for affected analytes for all associated samples with “E”.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
IS	Appropriate IS required for all analytes in all samples. Mass of IS must be <50 amu different from that of analyte	For each sample, IS intensity must be within 30-120% of that of initial calibration standard.	Reanalyze affected samples.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Per the method, for each sample, IS intensity must be ≥ 70% of that of initial calibration standard.

SAP Worksheet #28f – Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Soil/Sediment/ Water
Analytical Group	Metals (Mercury)
Analytical Method/ SOP Reference	SW-846 7470A, 7471B / CA-611, CA-615

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per digestion batch of 20 or fewer samples of similar matrix.	No mercury > ½ LOQ and > 1/10 the amount measured in any sample or 1/10 the PSL, whichever is greater. For negative blanks, absolute value < LOD.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	Analyst, Laboratory Department Manager and Data Validator	Bias/contamination	Same as Method/SOP QC Acceptance Limits.
LCS	One per digestion batch of 20 or fewer samples of similar matrix.	Water and Soil: %R must be within 80-120%.	Redigest and reanalyze all associated samples for affected analyte.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
MS (not applicable for rinsate blanks)	One per sample delivery group (SDG) or every 20 samples.	%R should be within 80-120% if sample < 4x spike added.	Flag results for affected analytes for all associated samples with "N".	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
Laboratory Duplicate (not applicable for rinsate blanks)	One per sample delivery group (SDG) or every 20 samples.	Project-specific criteria: If values are ≥ 5x LOQ, RPD should be ≤ 20%. If values are < 5x LOQ, Absolute Difference should be ≤ LOQ.	Flag results for affected analytes for all associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Precision	Waters: If values are ≥ 5x LOQ, RPD should be ≤ 20%; if values are < 5x LOQ, Absolute Difference should be ≤ LOQ. Soils: If values are ≥ 5x LOQ, RPD should be ≤ 35%; if values are < 5x LOQ, Absolute Difference should be ≤ 2x LOQ.

SAP Worksheet #28g -- QC Samples Table

(UFP-QAPP Manual Section 3.4)

Note: TestAmerica's statistically-derived QC limits referenced in the worksheet below refer to TestAmerica's limits at the time of analysis. TestAmerica's current limits are presented in Appendix E.

Matrix	Soil/Sediment/ Aqueous Field QC Samples
Analytical Group	Dioxins
Analytical Method/ SOP Reference	SW-846 8290/ WS-ID-0005

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
Method Blank	One per preparation batch	Project specific criteria, if available. Otherwise, no target analytes detected ≥ LOD or ≥ 20% of the associated regulatory limit or ≥ 5% of the sample result for the analyte, whichever is greater. (OCDD is considered a common laboratory contaminant and treated accordingly).	Verify instrument clean (evaluate calibration blank & samples prior to method blank), then reanalyze. Evaluate to determine if systematic issue within laboratory. Correct, then re-prepare and reanalyze the method blank and all samples processed with the contaminated blank in accordance with DoD QSM requirements. "Totals" are not considered "target analytes" – no corrective action or flagging is necessary for "totals".	Analyst, Laboratory Department Manager, and Data Validator	Bias/ contamination	Same as Method/SOP QC Acceptance Limits.
Internal Standard Spike	Every field sample, standard and QC sample	%R for each IS in the original sample (prior to dilutions) must be within 40-135% for all 2378-substituted internal standards.	Correct problem, then reprepare and reanalyze the samples with failed IS.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per sample preparation batch	%R must be within TestAmerica's statistically-derived control limits.	Reanalyze LCS once. If acceptable, report. Otherwise, evaluate and reprepare and reanalyze the LCS and all samples in the associated preparation batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Soil/Sediment/ Aqueous Field QC Samples
Analytical Group	Dioxins
Analytical Method/ SOP Reference	SW-846 8290/ WS-ID-0005

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria
MS/MSD (not applicable for rinsate blanks)	One MS/MSD per analytical/preparation batch	%R must be within TestAmerica's statistically-derived control limits; RPD must be ≤ 20%.	Identify problem; if not related to matrix interference, re-extract and reanalyze MS/MSD and all associated batch samples in accordance with DoD QSM requirements.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/ Bias/ Precision	Same as Method/SOP QC Acceptance Limits.

SAP Worksheet #28h – Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Water / Soil
Analytical Group	GRO
Analytical Method/ SOP Reference	SW-846 8015C / CA-316

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparation batch of twenty or fewer samples of similar matrix.	No analytes detected >1/2 the LOQ and >1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Investigate source of contamination. Evaluate the samples and associated QC: i.e., if the blank results are above the LOQ, report samples results which are < LOQ and >10X the blank. Otherwise, reprepare a blank and the remaining samples.	Analyst, Supervisor, QA Manager	Accuracy/Bias, Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogates	One per sample – BFB	%R must be within Katahdin's statistically-derived QC limits.	For QC and field samples, correct problem then reprepare and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepared within hold time.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparation batch of twenty or fewer samples of similar matrix.	%R must be within Katahdin's statistically-derived QC limits.	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was analyzed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are < LOQ, narrate. Otherwise, reprep a blank and the remaining samples.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Water / Soil
Analytical Group	GRO
Analytical Method/ SOP Reference	SW-846 8015C / CA-316

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
MS/MSD (not applicable for rinsate blanks)	One per SDG or every 20 samples.	%R should be within Katahdin's statistically-derived QC limits. Water and Soil Precision: RPD should be ≤ 30%.	Corrective actions will not be taken for samples when recoveries are outside limits if likely due to matrix, otherwise contact client.	Analyst, Supervisor, QA Manager	Precision/Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Worksheet #28i – Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix	Water / Soil
Analytical Group	ExTPH
Analytical Method/ SOP Reference	SW-846 8015C / CA-315

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparation batch of twenty or fewer samples of similar matrix.	No analytes detected >1/2 the LOQ and >1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	Investigate source of contamination. Evaluate the samples and associated QC: i.e., if the blank results are above the LOQ, report samples results which are < LOQ and >10X the blank. Otherwise, reprepare a blank and the remaining samples.	Analyst, Supervisor, QA Manager	Accuracy/Bias, Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogates	One per sample – Ortho-terphenyl	%R must be within Katahdin's statistically-derived QC limits.	For QC and field samples, correct problem then reprepare and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepared within hold time.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparation batch of twenty or fewer samples of similar matrix.	%R must be within Katahdin's statistically-derived QC limits.	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was analyzed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are < LOQ, narrate. Otherwise, reprepare a blank and the remaining samples.	Analyst, Supervisor, QA Manager	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

Matrix	Water / Soil
Analytical Group	ExTPH
Analytical Method/ SOP Reference	SW-846 8015C / CA-315

QC Sample:	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
MS/MSD (not applicable for rinsate blanks)	One per SDG or every 20 samples.	%R should be within Katahdin's statistically-derived QC limits. Water and Soil Precision: RPD should be ≤ 30%.	Corrective actions will not be taken for samples when recoveries are outside limits if likely due to matrix, otherwise contact client.	Analyst, Supervisor, QA Manager	Precision/Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.

SAP Worksheet #29 -- Project Documents and Records Table

(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
Field Documents Field Logbook Field Sample Forms Chain-of-Custody Records Air Bills Sampling Instrument Calibration Logs Sampling Notes Drilling Logs Photographs FTMR Forms SAP HASP	Field documents will be maintained in the project file located in the Tetra Tech Wilmington, Massachusetts office.
Laboratory Documents and Records - in the form of analytical data package: Sample receipt/login form Sample storage records Sample preparation logs Equipment calibration logs Sample analysis run logs Reported results for standards, QC checks, and QC samples Data completeness checklists Telephone logs Extraction/clean-up records Raw data	Laboratory documents will be included in the hardcopy and electronic deliverables from the laboratory. Laboratory data deliverables will be maintained in the Tetra Tech Wilmington, Massachusetts project file and in long-term data package storage at a third-party professional document storage firm. Electronic data results will be maintained in a database on a password protected Structured Query Language (SQL) server.
Assessment Findings Field Sampling Audit Checklist (if conducted) Analytical Audit Checklist (if conducted) Data Validation Memoranda (include tabulated data summary forms)	All assessment documents will be maintained in the Tetra Tech Wilmington, Massachusetts project file.
Reports Data Report	All versions of the Project Report and support documents (e.g., Data Validation Reports) will be stored in hard copy in the Tetra Tech Wilmington, Massachusetts project file and electronically in the server library.

SAP Worksheet #30 -- Analytical Services Table

(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ID Number	Analytical Method	Data Package Turnaround Time	Laboratory / Organization (name and address, contact person and telephone number)	Backup Laboratory / Organization (name and address, contact person and telephone number)
Soil, Sediment, and Aqueous	VOCs	See Worksheet #18	SW-846 8260B	21 days	Katahdin Analytical Services, Inc. 600 Technology Way Scarborough, Maine 04074 Contact: Ms. Kelly Perkins (207) 874-2400	Not applicable
	GRO and ExTPH		SW-846 8015C			
	PAHs		SW-846 8270D SIM			
	Metals		SW-846 6020A, 7470A, 7471B			
Aqueous	EDB		SW-846 8011			
Soil and Aqueous	PCBs		SW-846 8082A			
Soil, Sediment, and Aqueous	Dioxins	See Worksheet #18	SW-846 8290	21 days	TestAmerica 880 Riverside Parkway West Sacramento, CA 95605 Contact: Mr. Nilo Ligi 916-374-4427	Not applicable

Data packages will be provided as both hardcopy and portable document format (.PDF). Laboratories will provide a NIRIS compatible EDD. Data packages will be Contract Laboratory Program (CLP)-equivalent (i.e., they will contain CLP-equivalent summary forms and raw data). Data will be stored by the analytical laboratories for seven years.

SAP Worksheet #31 -- Planned Project Assessments Table

(*UFP-QAPP Manual Section 4.1.1*)

Assessment Type	**Frequency**	**Internal or External**	**Organization Performing Assessment**	**Person(s) Responsible for Performing Assessment** (title and organizational affiliation)	**Person(s) Responsible for Responding to Assessment Findings** (title and organizational affiliation)	**Person(s) Responsible for Identifying and Implementing Corrective Actions (CA)** (title and organizational affiliation)	**Person(s) Responsible for Monitoring Effectiveness of CA** (title and organizational affiliation)
Laboratory System Audit[1]	Every 2 years	External	DoD Environmental Laboratory Accreditation Program (ELAP) Accrediting Body	DoD ELAP Accrediting Body Auditor	Katahdin QAM TestAmerica QAM	Katahdin QAM TestAmerica QAM	DoD ELAP Accrediting Body Auditor

1. Katahdin and TestAmerica successfully completed the DoD's ELAP audit for all analytical methods. Copies of Katahdin's and TestAmerica's DoD ELAP accreditations are included in Appendix E.

SAP Worksheet #32 -- Assessment Findings and Corrective Action Responses

(*UFP-QAPP Manual Section 4.1.2*)

Assessment Type	**Nature of Deficiencies Documentation**	**Individual(s) Notified of Findings** (name, title, organization)	**Timeframe of Notification**	**Nature of Corrective Action Response Documentation**	**Individual(s) Receiving Corrective Action Response** (name, title, organization)	**Timeframe for Response**
Laboratory System Audit	Written audit report	QAM, Katahdin QAM, TestAmerica	Not specified by DoD ELAP	Letter	DoD ELAP Accrediting Body	Specified by DoD ELAP

SAP Worksheet #33 -- QA Management Reports Table

(*UFP QAPP Manual Section 4.2)*

Type of Report	**Frequency** (daily, weekly monthly, quarterly, annually, etc.)	**Projected Delivery Date(s)**	**Person(s) Responsible for Report Preparation** (title and organizational affiliation)	**Report Recipient(s)** (title and organizational affiliation)
Data validation report	Per SDG	Within 3 weeks of receipt of laboratory data	Project Chemist, Tetra Tech	PM, Tetra Tech Tetra Tech project file
Major analysis problem identification (internal memorandum)	When persistent analysis problems are detected	Immediately on detection of problem (same day)	QAM, Tetra Tech	PM (Tetra Tech), Program Manager (Tetra Tech), Tetra Tech project file
Project monthly progress report[1]	Monthly for duration of the project	Monthly	PM, Tetra Tech	Navy, project file
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately on detection of problem (same day)	PM, Katahdin PM, TestAmerica	Tetra Tech project file

1. The monthly progress report is an update for the Navy RPM and contract office. The report includes information such as activities completed, an updated schedule, identification of outstanding issues, plans for the next period, and a financial narrative.

SAP Worksheet #34 -- Verification (Step I) Process Table
(*UFP-QAPP Manual Section 5.2.1*)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-Custody Forms	The Tetra Tech FOL or designee will review and sign the chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and the sample information is accurate. The forms will be signed by the sampler and a copy will be retained for the project file, the Tetra Tech PM, and the Tetra Tech Data Validators.	Internal	Sampler and FOL, Tetra Tech
	The Laboratory Sample Custodian will review the sample shipment for completeness, integrity, and sign accepting the shipment. The Tetra Tech Data Validators will check that the chain-of-custody form was signed/dated by the Tetra Tech FOL or designee relinquishing the samples and also by the Laboratory Sample Custodian receiving the samples for analyses.	Internal/ External	1 - Laboratory Sample Custodian, Katahdin and TestAmerica 2 - Data Validators, Tetra Tech
SAP Sample Tables/ Chain-of-Custody Forms	Verify that all proposed samples listed in the SAP tables have been collected.	Internal	FOL or designee, Tetra Tech
Sample Log Sheets	Verify that information recorded in the log sheets is accurate and complete.	Internal	FOL or designee, Tetra Tech
Sample coordinates	Verify that actual sample locations are correct and in accordance with the SAP proposed locations. Document any discrepancies in the final report.	Internal	Tetra Tech, FOL or designee
SAP/ Field Logs/ Analytical Data Packages	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and MPCs have been achieved. Particular attention should be given to verify that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken trail of documented chain-of-custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the sampling plan was implemented and carried out as written and that any deviations are documented.	Internal	PM or designee, Tetra Tech
SAP/ Laboratory SOPs/ Raw Data/ Applicable Control Limits Tables	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied. Establish that all method QC samples were analyzed and in control as listed in the analytical SOPs. If method QA is significantly out of control, the Laboratory QAM will contact the Tetra Tech PM via telephone or e-mail for guidance prior to report preparation.	Internal	Laboratory QAM, Katahdin and TestAmerica

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
SAP/ Chain-of-Custody Forms	Check that field QC samples listed in Worksheet #20 were collected as required.	Internal	FOL or designee, Tetra Tech
Analytical Data Packages	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The Laboratory QAM will sign the case narrative for each data package.	Internal	Laboratory QAM, Katahdin and TestAmerica
EDDs/ Analytical Data Packages	Each EDD will be verified against the chain-of-custody and hard copy data package for accuracy and completeness. Laboratory analytical results will be verified and compared to the electronic analytical results for accuracy. Sample results will be evaluated for laboratory contamination and will be qualified for false positives using the laboratory method/preparation blank summaries. Positive results reported between the DL and the LOQ will be qualified as estimated. Extraneous laboratory qualifiers will be removed from the validation qualifier.	External	Data Validators, Tetra Tech
	Each data package will be verified for completeness by the Tetra Tech Data Validator. Missing information will be requested by the Tetra Tech Data Validator from the Laboratory PM.	External	Data Validators, Tetra Tech

SAP Worksheet #35 -- Validation (Steps IIa and IIb) Process Table
(UFP-QAPP Manual Section 5.2.2) (Figure 37 UFP-QAPP Manual) (Table 9 UFP-QAPP Manual)

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIa	Chain-of-Custody Forms	Custody - Ensure that the custody and integrity of the samples was maintained from collection to analysis and the custody records are complete and any deviations are recorded. Review that the samples were shipped and store at the required temperature and sample pH for chemically-preserved samples meet the requirements listed in Worksheet #19. Ensure that the analyses were performed within the holding times listed in Worksheet #19.	Project Chemist or Data Validators, Tetra Tech
IIa/IIb	SAP/ Laboratory Data Packages/ EDDs	Ensure that the laboratory QC samples listed in Worksheet #28 were analyzed and that the MPCs listed in Worksheet #12 were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Project Chemist or Data Validators, Tetra Tech
		Check the field sampling precision by calculating the RPD for field duplicate samples. Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSDs; and LCS/ LCSD, if available. Ensure compliance with the methods and project MPCs accuracy goals listed in Worksheet #12.	
		Check that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis.	
		Review the chain-of-custody forms generated in the field to ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. The Tetra Tech Data Validator will verify that elements of the data package required for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per Worksheet #36. Check that all data have been transferred correctly and completely to the final SQL database.	

SAP Worksheet #35 – Validation (Steps IIa and IIb) Process Table (Continued)

Step IIa / IIb	Validation Input	Description	Responsible for Validation (name, organization)
IIb	SAP/ Laboratory Data Packages/ EDDs		Project Chemist or Data Validators, Tetra Tech
		QA/QC - Ensure that all QC samples specified in the SAP were collected and analyzed and that the associated results were within prescribed SAP acceptance limits. Ensure that QC samples and standards prescribed in analytical SOPs were analyzed and within the prescribed control limits. If any significant QC deviations occur, the Laboratory QAM shall have contacted the Tetra Tech PM.	
		Deviations - Summarize deviations from methods, procedures, or contracts in the Data Validation Report. Determine the impact of any deviation from sampling or analytical methods and SOPs requirements and matrix interferences effect on the analytical results. Qualify data results based on method or QC deviation and explain all the data qualifications. Print a copy of the project database qualified data depicting data qualifiers and data qualifiers codes that summarize the reason for data qualifications. Determine if the data met the MPCs and discuss the potential impact of any deviations on the technical usability of the data.	

SAP Worksheet #36 -- Validation (Steps IIa and IIb) Summary Table
(*UFP-QAPP Manual Section 5.2.2.1*)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	**Data Validator** (title and organizational affiliation)
IIa and IIb	Soil, Sediment, and Aqueous	VOCs, PAHs, GRO, ExTPH	Tier II(1) data validation. Project-specific criteria for VOCs by SW-846 8260B PAHs by SW-846 8270D SIM, and GRO and ExTPH by SW-846 8015C are listed in Worksheets #12, #15, #19, #24, and #28. Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part II, December 1996 (USEPA, 1996b) will be applied using these criteria.	Tetra Tech, Project Chemist (K. Carper) and staff chemists
IIa and IIb	Soil and Aqueous	PCBs and EDB	Tier II[1] data validation. Criteria for PCBs by SW-846 8082A and for EDB by SW-846 8011 are listed in Worksheets #12, #15, #19, #24, and #28. Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part III, February 2004 (EPA, 2004) will be applied using these criteria.	
IIa and IIb	Soil, Sediment, and Aqueous	Dioxins	Tier II(1) data validation. Project-specific criteria for dioxins by SW-846 8290 are listed in Worksheets #12, #15, #19, #24, and #28. USEPA National Functional Guidelines for Chlorinated Dibenzo-p-Dioxins (CDDs) and Chlorinated Dibenzofurans (CDFs) Data Review, September 2005 (EPA, 2005b) will be applied using these criteria.	
IIa and IIb	Soil, Sediment, and Aqueous	Metals	Tier II(1) data validation. Project-specific criteria for metals by SW-846 6020A/7470A/7471B are listed in Worksheets #12, #15, #19, #24, and #28. Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part IV, November 2008 (USEPA, 2008) will be applied using these criteria.	

1 – As defined in the Region I EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analyses, Part I, Attachment B, "Region 1 Tiered Organic and Inorganic Data Validation Guidelines", July 1, 1993, Draft (USEPA, 1996b).

SAP Worksheet #37 -- Usability Assessment

(*UFP-QAPP Manual Section 5.2.3*)

Data Usability Assessment

The usability of the data directly affects whether project objectives can be achieved. The following characteristics will be evaluated at a minimum. The results of these evaluations will be included in the project report. The characteristics will be evaluated for multiple concentration levels if the evaluator determines that this is necessary. To the extent required by the type of data being reviewed, the assessors will consult with other technically competent individuals to render sound technical assessments of these data characteristics:

Completeness: The FOL, acting on behalf of the Project Team, will determine whether deviations from the scheduled sample collection or analyses occurred. If they have occurred and the Tetra Tech PM determines that the deviations compromise the ability to meet project objectives she will consult with the Navy RPM and other project team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions.

Precision: The Project Chemist, or designee, acting on behalf of the Project Team, will determine whether precision goals for field duplicates and laboratory duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in Worksheets #12 and #28. This will also include a comparison of field and laboratory precision with the expectation that laboratory duplicate results will be no less precise than field duplicate results. If the goals are not met, or data have been flagged as estimated (J qualifier), limitations on the use of the data will be described in the project report.

Accuracy: The Project Chemist, acting on behalf of the Project Team, will determine whether the accuracy/bias goals were met for project data. This will be accomplished by comparing percent recoveries of LCS, LCSD, MS, MSD, and surrogate compounds to accuracy goals identified in Worksheet #28. This assessment will include an evaluation of field and laboratory contamination; instrument calibration variability; and analyte recoveries for surrogates, matrix spike, matrix spike duplicate, and laboratory control samples. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

Representativeness: A project scientist identified by the Tetra Tech PM, and acting on behalf of the Project Team, will determine whether the data are adequately representative of intended populations, both spatially and temporally. This will be accomplished by verifying that samples were collected and analyzed in accordance with this SAP, by reviewing spatial and temporal data variations, and by comparing these characteristics to expectations. The usability report will describe the representativeness of the data for each matrix and analytical fraction. This will not require quantitative comparisons unless professional judgment of the project scientist indicates that a quantitative analysis is required.

Comparability: The PM or designee, acting on behalf of the Project Team, will determine whether the data generated under this project are sufficiently comparable to historical property data generated by different methods and for samples collected using different procedures and under different property conditions. This will be accomplished by comparing overall precision and bias among data sets for each matrix and analytical fraction. This will not require quantitative comparisons unless the Project Chemist indicates that such quantitative analysis is required.

Sensitivity: The Project Chemist, acting on behalf of the Project Team, will determine whether project sensitivity goals listed in Worksheet #15 are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described.

Data Usability Assessment

Describe the evaluative procedures used to assess overall measurement error associated with the project:

After completion of the data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. In addition to the evaluations described above, a series of inspections and statistical analyses will be performed to estimate these characteristics. The statistical evaluations will include simple summary statistics for target analytes, such as maximum concentration, minimum concentration, number of samples exhibiting non-detected results, number of samples exhibiting positive results, and the proportion of samples with detected and non-detected results. The Project Team members identified by the PM will assess whether the data collectively support the attainment of project objectives. They will consider whether any missing or rejected data have compromised the ability to make decisions or to make the decisions with the desired level of confidence. The data will be evaluated to determine whether missing or rejected data can be compensated by other data.

For statistical evaluations, Worksheet #11 describes how to treat non–detected values.

Identify the personnel responsible for performing the usability assessment:

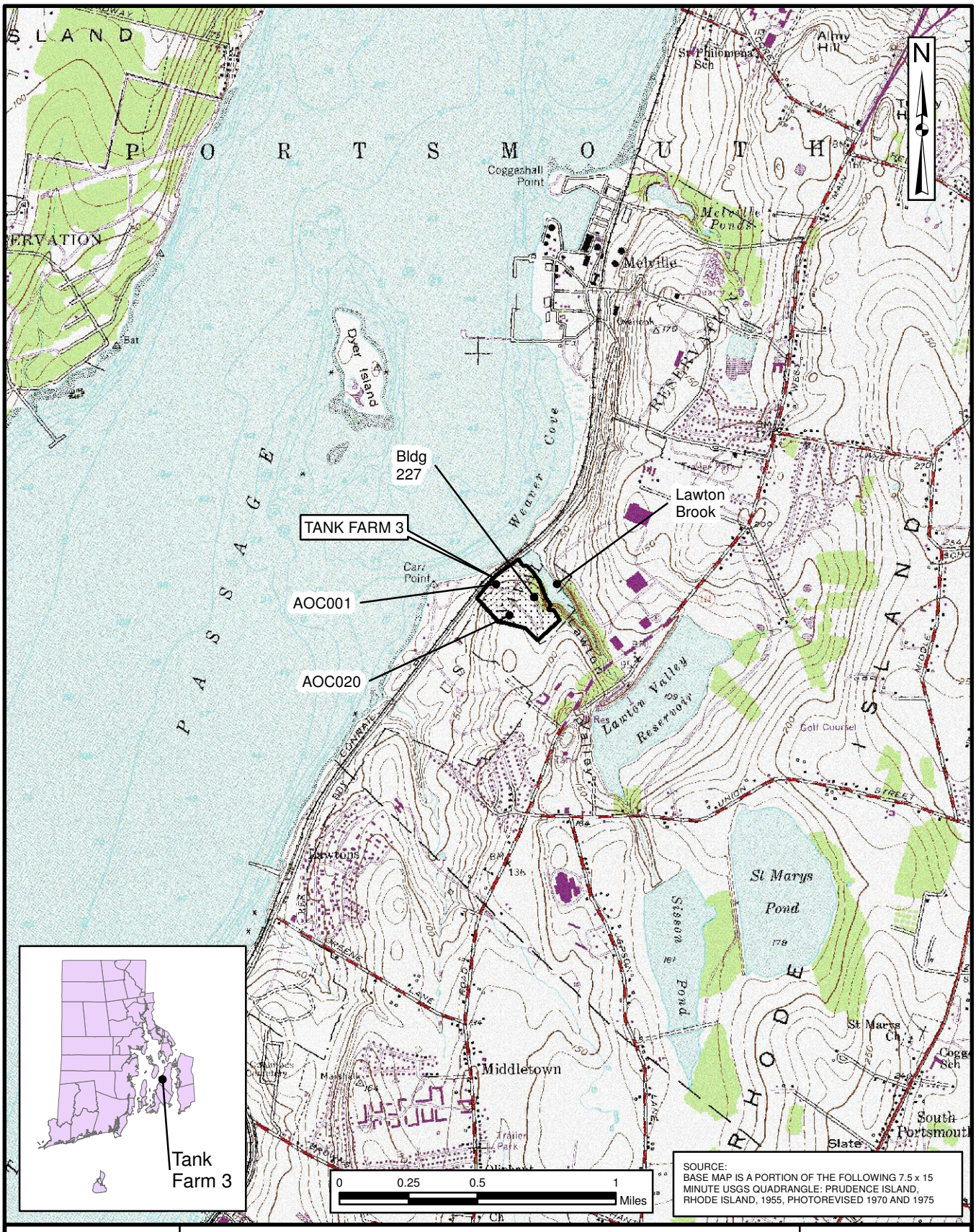
The Tetra Tech PM, Project Chemist, and FOL will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed with the Project Team. If deficiencies affecting the attainment of project objectives are identified, the review will take place either in a face to face meeting or a teleconference depending on the extent of identified deficiencies. If no significant deficiencies are identified, the data usability assessment will simply be documented in the project report and reviewed during the normal document review cycle.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies:

The data will be presented in tabular format, including data qualifications such as estimation (J, UJ) or rejection (R). The project report will identify and describe the data usability limitations and suggest re-sampling or other corrective actions, if necessary. Graphical presentations of the data such as concentration tag maps will be generated as part of the overall data evaluation process.

FIGURES

I:\02710\PP.DF\TF3_LOCUS.MXD DWM 09/17/12



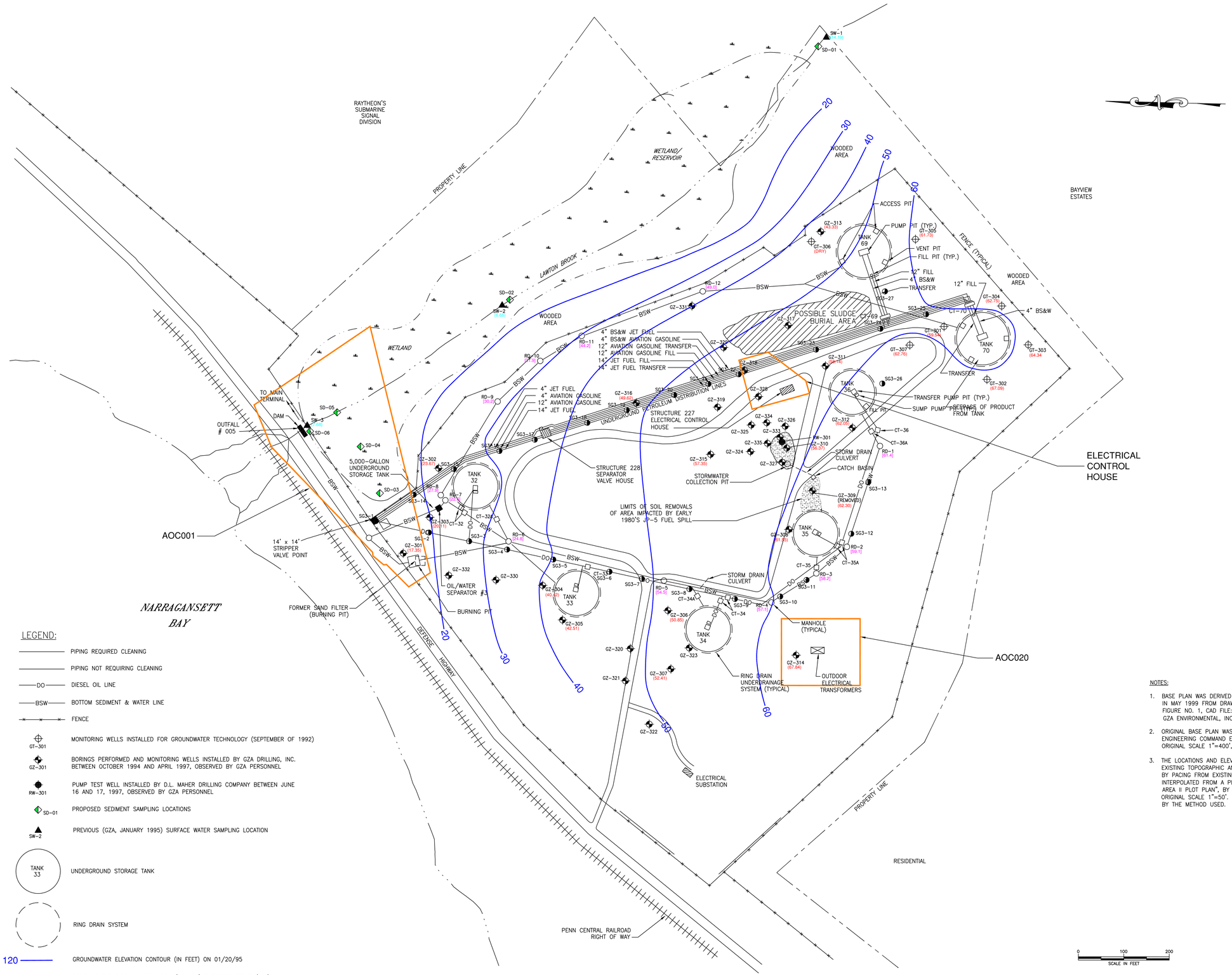
TETRA TECH

NAVSTA NEWPORT
PORTSMOUTH, RHODE ISLAND

SITE LOCUS MAP

TANK FARM 3
CATEGORY 1 DATA GAPS ASSESSMENT
SAMPLING AND ANALYSIS PLAN

SCALE	AS NOTED
FILE	\..\TF3_LOCUS.MXD
REV	0
DATE	09/17/12
FIGURE NUMBER	FIGURE NO. 1



LEGEND:

- PIPING REQUIRED CLEANING
- PIPING NOT REQUIRING CLEANING
- DO — DIESEL OIL LINE
- BSW — BOTTOM SEDIMENT & WATER LINE
- FENCE
- GT-301 — MONITORING WELLS INSTALLED FOR GROUNDWATER TECHNOLOGY (SEPTEMBER OF 1992)
- GZ-301 — BORINGS PERFORMED AND MONITORING WELLS INSTALLED BY GZA DRILLING, INC. BETWEEN OCTOBER 1994 AND APRIL 1997, OBSERVED BY GZA PERSONNEL
- RW-301 — PUMP TEST WELL INSTALLED BY D.L. MAHER DRILLING COMPANY BETWEEN JUNE 16 AND 17, 1997, OBSERVED BY GZA PERSONNEL
- SD-01 — PROPOSED SEDIMENT SAMPLING LOCATIONS
- SW-2 — PREVIOUS (GZA, JANUARY 1995) SURFACE WATER SAMPLING LOCATION
- TANK 33 — UNDERGROUND STORAGE TANK
- RING DRAIN SYSTEM
- 120 — GROUNDWATER ELEVATION CONTOUR (IN FEET) ON 01/20/95
- (142.59) — INDICATES GROUNDWATER ELEVATION (IN FEET) RECORDED ON 01/20/95
- [61.4] — INDICATES GROUNDWATER ELEVATION (IN FEET) RECORDED IN RING DRAIN MANHOLE ON 01/20/95
- (7.68) — INDICATES SURFACE WATER ELEVATION (IN FEET) RECORDED ON 01/26/95

NOTES:

1. BASE PLAN WAS DERIVED AND DIGITIZED BY FOSTER WHEELER ENVIRONMENTAL CORPORATION IN MAY 1999 FROM DRAWING ENTITLED "DFSC/TANK FARM 3 – SITE PLAN", PROJECT NO. 31288.9, FIGURE NO. 1, CAD FILE: TANK-3.DWG, VIEW TANK3-LARGE, DATED JANUARY 1998, BY GZA ENVIRONMENTAL, INC.
2. ORIGINAL BASE PLAN WAS DEVELOPED FROM A PLAN PROVIDED BY NAVAL FACILITIES ENGINEERING COMMAND ENTITLED "NAVAL COMPLEX NEWPORT, R.I. MELVILLE (SOUTH)", ORIGINAL SCALE 1"=400', DRAWING NO. 944208.
3. THE LOCATIONS AND ELEVATIONS OF THE BORINGS WERE APPROXIMATELY DETERMINED BY SURVEY FROM EXISTING TOPOGRAPHIC AND MAN-MADE FEATURES. THE LOCATIONS OF THE TANKS WERE DETERMINED BY PACING FROM EXISTING TOPOGRAPHIC FEATURES. THE LOCATIONS OF THE UTILITY LINES WERE INTERPOLATED FROM A PLAN ENTITLED "FUEL DISTRIBUTION SYSTEM STORAGE TANKS 32-36, 69 & 70 AREA II PLOT PLAN", BY J.M. McCUSKER CO., DATED JUNE 11, 1954, DRAWING NO. Y&D# 627332, ORIGINAL SCALE 1"=50'. THIS DATA SHOULD BE CONSIDERED ACCURATE ONLY TO THE DEGREE IMPLIED BY THE METHOD USED.

TETRA TECH

NAVSTA NEWPORT
PORTSMOUTH, RHODE ISLAND

SITE PLAN AND GROUNDWATER CONTOURS (01/20/95)

TANK FARM 3
CATEGORY 1 DATA GAPS ASSESSMENT
SAMPLING AND ANALYSIS PLAN

FILE	SCALE
\..\TF3_GW_CONTOUR_012095.DWG	AS NOTED
FIGURE NUMBER	**REV** / **DATE**
FIGURE NO. 2	0 / 5/21/13

ACAD:I:\02710\PP.FF\TF3_GW_CONTOUR_012095.DWG 5/21/13 DWM

I:\02710\PP-FF\TF3_AOC1_SAMP_LOCS.MXD DWMNEC 05/21/13

Legend

- Proposed Shallow Sediment Sample Location
- Proposed Shallow/Deep Sediment Sample/Boring Location
- Proposed Soil Boring Location
- Previous Soil Sample Location (TtEC, 2005)
- Monitoring Well Location
- Previous Soil Gas Sample Location (May 1997)
- Limit of Excavation
- Approximate AOC Location
- Test Pit Location
- Bottom Sediment & Water Line
- Discharge Line Shown on 1957 Plan
- Underground Petroleum Distribution Line
- Railroad
- Fenceline
- Wetland/Reservoir

N

SD-06
SD-05
SD-04
SD-03
SG3-1
SG3-14
SG3-2
GZ-301
GZ-303
GZ-302
TF3-001-SB-108
TF3-001-SB-107
TF3-001-SB-106
TF3-001-S2-BP
TF3-001-S1-2.0A
TF3-001-SB-105
TF3-001-SB-104
TF3-001-S1-2.0
TF3-001-SB-102
TF3-001-SB-103
TF3-001-S4-2.0
TF3-001-SB-101
TF3-001-S4-Bott
TF3-001-S3-2.5
AOC 1
OWS #3

Notes:
1. Locations approximate and based upon a combination of site plans and GPS coordinates obtained from the SIRAR (TtEC, 2005).
2. Not to be used for design.

0 20 40 80 Feet

TETRA TECH

NAVSTA NEWPORT
PORTSMOUTH, RHODE ISLAND

AOC 001

TANK FARM 3
CATEGORY 1 DATA GAPS ASSESSMENT
SAMPLING AND ANALYSIS PLAN

FILE	SCALE
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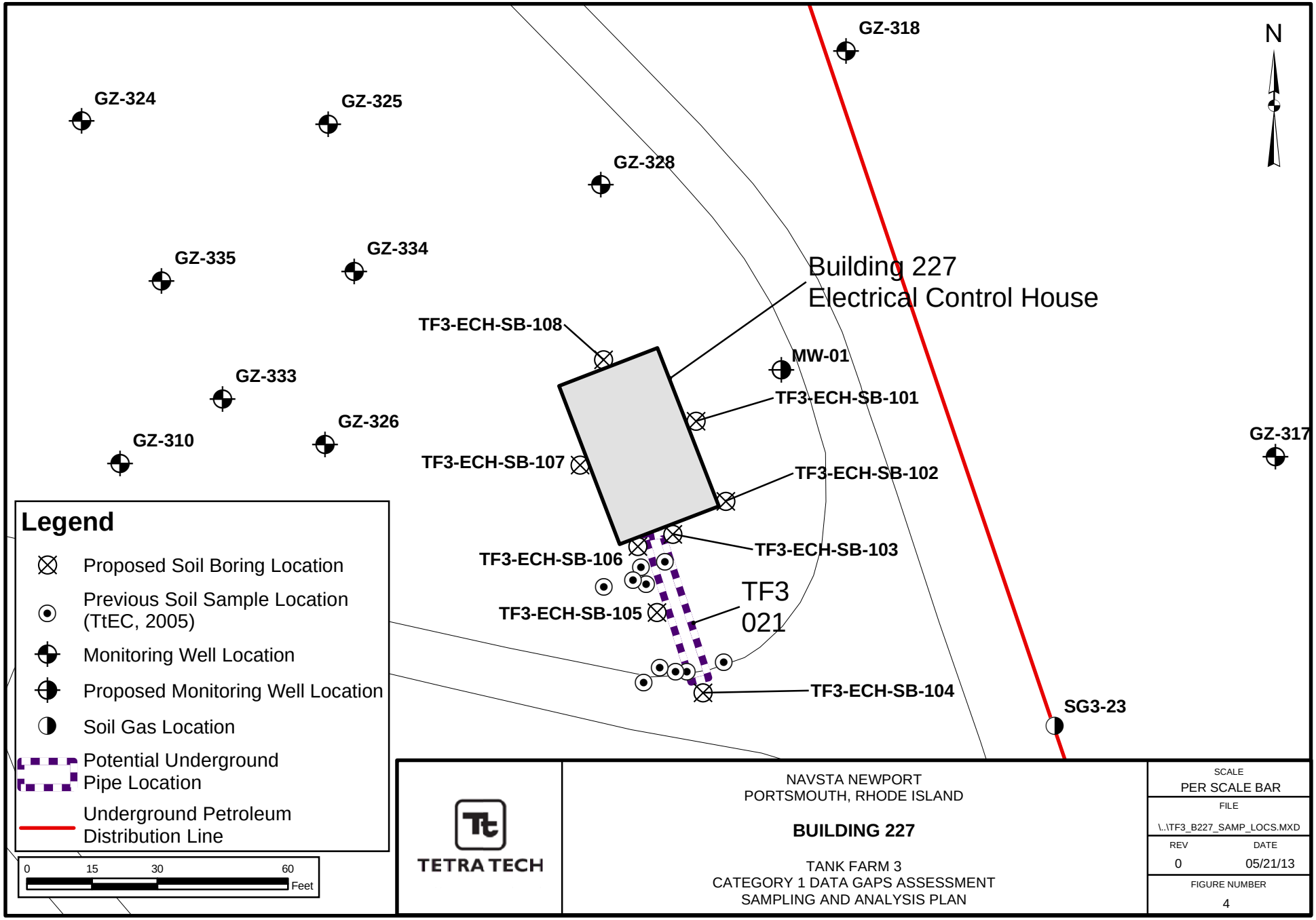

TETRA TECH

NAVSTA NEWPORT
PORTSMOUTH, RHODE ISLAND

BUILDING 227

TANK FARM 3
CATEGORY 1 DATA GAPS ASSESSMENT
SAMPLING AND ANALYSIS PLAN

SCALE	
PER SCALE BAR	
FILE	
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REV	DATE
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FIGURE NUMBER	
4	

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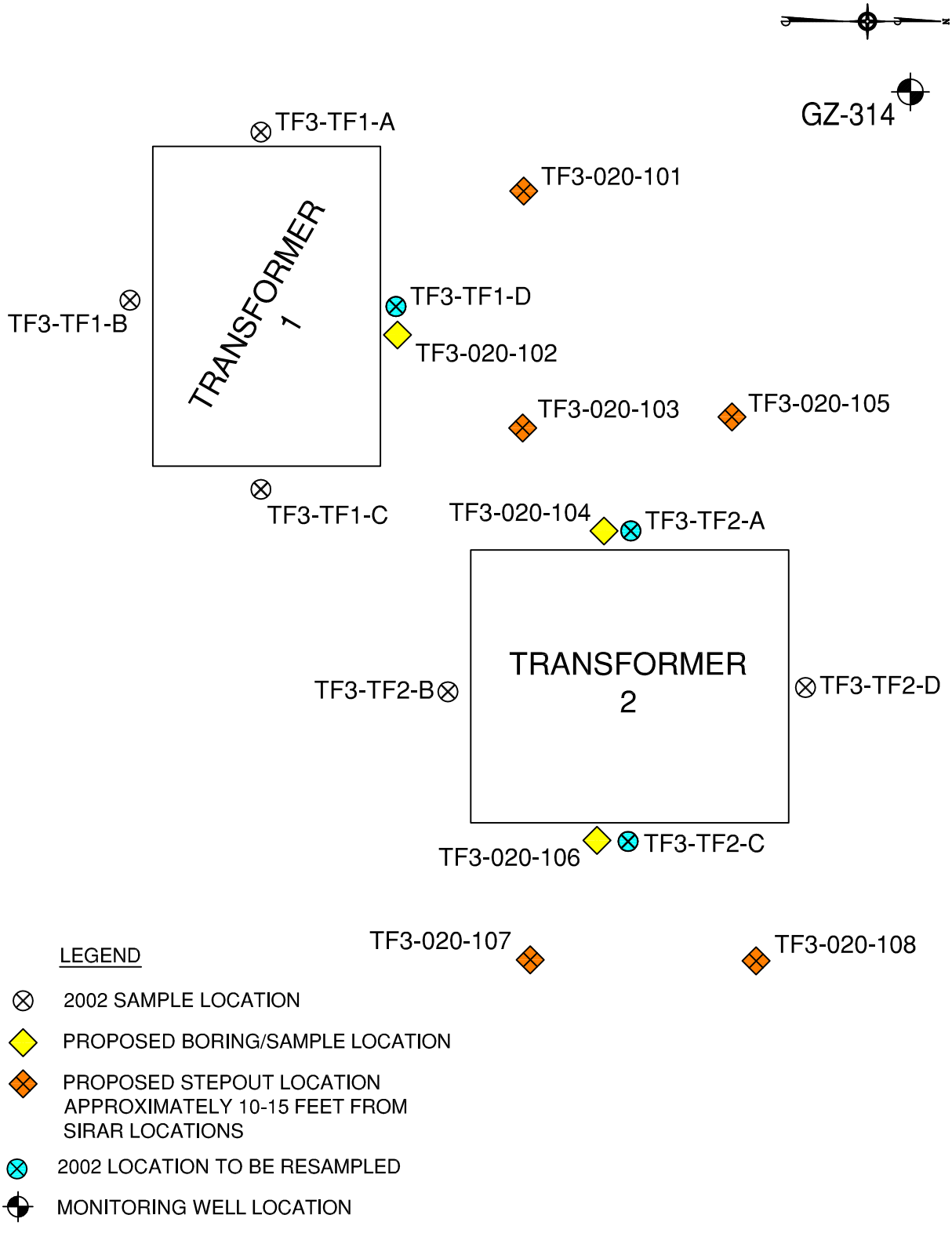

TETRA TECH

NAVSTA NEWPORT
PORTSMOUTH, RHODE ISLAND

SCHEMATIC OF AOC-020

TANK FARM 3
CATEGORY 1 DATA GAPS ASSESSMENT
SAMPLING AND ANALYSIS PLAN

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REV	0
DATE	9/17/12
FIGURE NUMBER	FIGURE NO. 5

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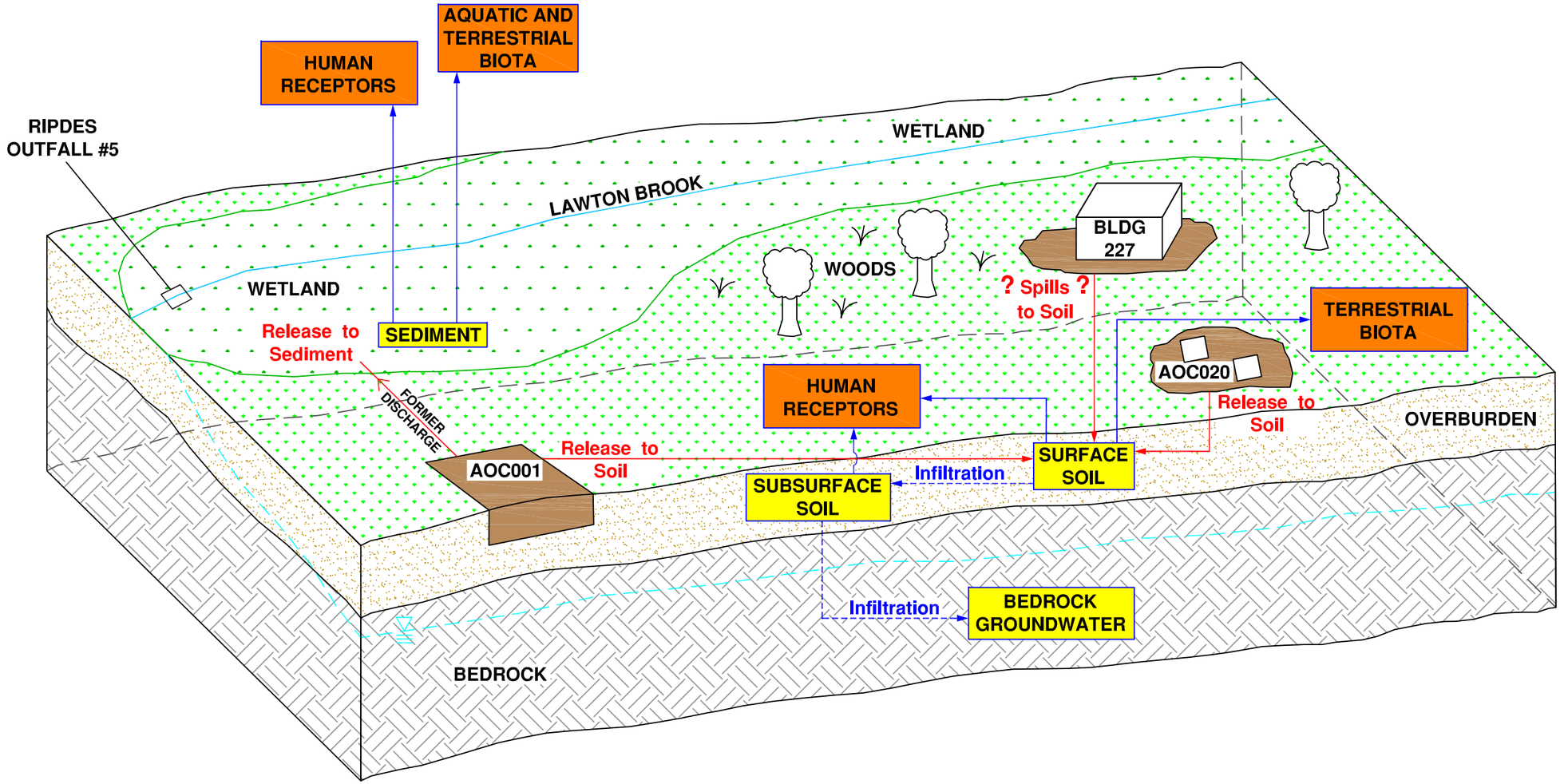

LEGEND

- POTENTIAL EXISTING RECEPTORS
- POTENTIALLY AFFECTED MEDIA
- GROUNDWATER TABLE

RED TEXT/ARROWS = CONTAMINANT SOURCE/DISCHARGE ROUTE

BLUE TEXT/ARROWS = FATE AND TRANSPORT ROUTE

NOTE: RELEASE TO AIR FROM BURNING AT AOC001 LIKELY NOT TO CONTRIBUTE TO RISK.

TETRA TECH

NAVSTA NEWPORT
PORTSMOUTH, RHODE ISLAND

CONCEPTUAL SITE MODEL

TANK FARM 3
CATEGORY 1 DATA GAPS ASSESSMENT
SAMPLING AND ANALYSIS PLAN

FILE	SCALE
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FIGURE NUMBER: FIGURE NO. 6	REV: 0 DATE: 9/17/12

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APPENDIX A

SUMMARY OF RESULTS BY AREA AND CATEGORY

APPENDIX A
SUMMARY OF RESULTS BY AREA AND CATEGORY
TANK FARM 3- STUDY AREA SCREENING EVALUATION
NAVSTA NEWPORT, NEWPORT RI

Area	History	Status	Analytical Summary	Recommendations
Catogory 1 Area				
Burn (Sand Filter) Pit AOC 001	Tank bottoms and ring-drain water was historically pumped this sand filter. Residual oil remaining in the sand filter was reportedly burned or scraped off and removed to an off-site location.	In 2004, contaminated soil within pit and accessible contaminated soil outside pit was excavated. Inside of pit was pressure washed. The structure remains beneath six feet of clean fill.	Groundwater below criteria. Limited volume of soil with TPH above RIDEM criteria remain.	Due to sludge burning, further investigation under CERCLA and Navy IR Program.
Building 227	Electrical Control House	A transformer is located in this building and it potentially stored other contaminant sources.	One soil boring has been advanced downgradient of the building. Soil samples were collected and groundwater samples were collected. No evidence of contamination.	Further investigate the soils and groundwater in the vicinty of Building 227 under CERCLA and Navy IR Program.
AOC 020	2 Pad mounted transformers located within this AOC which consists of a former (decomissioned) tranformer blockhouse	Two pad monted transformers remain in this area. Soil sampling associated with the transformers occurred in 2004 and analysis for PCBs was performed.	Soil analytical results were below the RIDEM criteria. But were above the ORNL for PCBs.	Further investigate soil and groundwater for PCBs under CERCLA and Navy IR Program.
Category 2 Areas				
AOC 004	area of ground staining seen in aerial photograph in middle of loop road	test pits and soil remediation in 2004	Soil concentrations of TPH remain above RIDEM Residential and Industrial DEC	Further investigate/manage under the RIDEM UST program
AOC 017	discolored area along bank of drainage ditch seen in aerial photograph	test pits and soil remediation in 2004	PAHs in soil remaing above RIDEM Residential DEC.	Further investigate/manage under the RIDEM UST program
AOC 023	Ground staining from Tank 33 access vault	test pits and soil remediation in 2004	TPH in soil remaining above RIDEM Residential DEC.	Further investigate/manage under the RIDEM UST program
Tank 33	UST used for storage of fuels from World War 2 until the 1970s	Tank Closed	Downgradient monitoring well GZ-304 contained elevated levels of naphthalene in 2004	Further investigate/manage under the RIDEM UST program
Tank 32 Vent	Vent associated with UST used for storage of fuels from World War 2 until the 1970s	Tank Closed	TPH in soil remaining above RIDEM Residential DEC.	Manage with a CAP and LUC under the RIDEM UST program
AOC 005	discolored area seen in aerial photograph	test pits and soil remediation in 2004	TPH in soil remaining above RIDEM Residential DEC.	Manage with a CAP and LUC under the RIDEM UST program
AOC 009	discolored area seen in aerial photograph	test pits and soil remediation in 2004	TPH in soil remaining above RIDEM Residential DEC.	Manage with a CAP and LUC under the RIDEM UST program
AOC 010	discolored area seen in aerial photograph	test pits and soil remediation in 2004	TPH in soil remaining above RIDEM Residential DEC.	Manage with a CAP and LUC under the RIDEM UST program
AOC 012	possibly excavated area and staining seen in aerial photograph	test pits and soil remediation in 2004	TPH in soil remaining above RIDEM Residential DEC.	Manage with a CAP and LUC under the RIDEM UST program
AOC 018	disturbed vegetation seen in aerial photograph	test pits and soil remediation in 2004	TPH in soil remaining above RIDEM Residential DEC.	Manage with a CAP and LUC under the RIDEM UST program
AOC 029	staining and distressed vegetation seen in aerial photograph	test pits and soil remediation in 2004	TPH in soil remaining above RIDEM Residential DEC.	Manage with a CAP and LUC under the RIDEM UST program

APPENDIX A
SUMMARY OF RESULTS BY AREA AND CATEGORY
TANK FARM 3- STUDY AREA SCREENING EVALUATION
NAVSTA NEWPORT, NEWPORT RI

Area	History	Status	Analytical Summary	Recommendations
Category 3 Areas				
Building 228	potential floor drains withhin the valve house. The building contain(ed) possible sources	previously investigated for petroleum impacts, pipes were excavated, eight samples collected	no exceedences of RDEC for TPH	Investigate the inside of the building and develop a SAP if a floor drain is found
Electrical substation	electrical substation in the southewest corner of property	not investigated	not applicable	Further evaluation to determine if additional investigation is required
Former Building 108	location of former pump house/ security shed. Housed switch gear to activate the fire suppression system	Building no longer exists. Not investigated	not applicable	Further evaluation to determine if additional investigation is required
Building 229	A stripper valve point, part of petroleum distribution piping	Previously cleaned and decommissioned. No record of release.	not applicable	Further evaluation to determine if additional investigation is required
Possible OWS#4	A structure was labelled OWS#4 in the Work Plan for Site Closure.	There are no records of this OWS and it was likely never built or was mis-labelled	not applicable	Further evaluation to determine if this structure exists and if so, if an investigation is required
Fencing	Painted metal fence around the Site	not investigated	not applicable	Further evaluation to determine if additional investigation is required

APPENDIX B

TETRA TECH AND EPA SOPs

TETRA TECH NUS, INC.	STANDARD OPERATING PROCEDURES	Number CT-05	Page 1 of 7
		Effective Date 01/29/01	Revision 2
		Applicability Tetra Tech NUS, Inc.	
		Prepared Management Information Systems Department	
Subject DATABASE RECORDS AND QUALITY ASSURANCE		Approved D. Senovich	

TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this document is to specify a consistent procedure for the quality assurance review of electronic and hard copy databases. This SOP outlines the requirements for establishment of a Database Record File, Quality Assurance review procedures, and documentation of the Quality Assurance Review Process.

2.0 SCOPE

The methods described in this Standard Operating Procedure (SOP) shall be used consistently for all projects managed by Tetra Tech NUS (TtNUS).

3.0 GLOSSARY

Chain-of-Custody Form - A Chain-of-Custody Form is a printed form that accompanies a sample or a group of samples from the time of sample collection to the laboratory. The Chain-of-Custody Form is retained with the samples during transfer of samples from one custodian to another. The Chain-of-Custody Form is a controlled document that becomes part of the permanent project file. Chain-of-Custody and field documentation requirements are addressed in SOP SA-6.1.

Electronic Database - A database provided on a compact laser disk (CD). Such electronic databases will generally be prepared using public domain software such as DBase, RBase, Oracle, Visual FoxPro, Microsoft Access, Paradox, etc.

Hardcopy Database - A printed copy of a database prepared using the software discussed under the definition of an electronic database.

Form I - A printed copy of the analytical results for each sample.

Sample Tracking Summary - A printed record of sample information including the date the samples were collected, the number of samples collected, the sample matrix, the laboratory to which the samples were shipped, the associated analytical requirements for the samples, the date the analytical data were received from the laboratory, and the date that validation of the sample data was completed.

4.0 RESPONSIBILITIES

Database Records Custodian - It shall be the responsibility of the Database Records Custodian to update and file the Sample Tracking Summaries for all active projects on a weekly basis. It shall be the responsibility of the Database Records Custodian to ensure that the most recent copies of the Sample Tracking Summaries are placed in the Database Records file. It shall be the responsibility of the Database Records Custodian to ensure that a copy of all validation deliverables is provided to the Project Manager (for placement in the project file). It shall be the responsibility of the Database Records Custodian to ensure that photocopies of all validation deliverables and historical data and reports (as applicable) are placed in the Database Records file.

Data Validation Coordinator - It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that the Sample Tracking Summaries are maintained by the Database Records Custodian. It shall be the responsibility of the Data Validation Coordinator (or designee) to ensure that photocopies of all data validation deliverables are placed in the applicable Database Records file by the Database Records Custodian.

Earth Sciences Department Manager - It shall be the responsibility of the Earth Sciences Department Manager (or equivalent) to ensure that all field personnel are familiar with the requirements of this Standard Operating Procedure (specifically Section 5.5).

FOL - It shall be the responsibility of the FOL (FOL) of each project to ensure that all field technicians or sampling personnel are thoroughly familiar with this SOP, specifically regarding provision of the Chain-of-Custody Forms to the Database Records Custodian. Other responsibilities of the FOL are described in Sections 5.4 and 5.5.

Management Information Systems (MIS) Manager - It shall be the responsibility of the MIS Manager to ensure that copies of original electronic deliverables (CDs) are placed in both the project files and the Database Records File. It shall be the responsibility of the MIS Manager (or designee) to verify the completeness of the database (presence of all samples) in both electronic and hardcopy form in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that Quality Assurance Reviews are completed and are attested to by Quality Assurance Reviewers. It shall be the responsibility of the MIS Manager to ensure that records of the Quality Assurance review process are placed in the Database Records File. It shall be the responsibility of the MIS Manager to ensure that both electronic and hardcopy forms of the final database are placed in both the project and the Database Record File. It shall be the responsibility of the MIS Manager to ensure that data validation qualifiers are entered in the database.

Furthermore, it shall be the responsibility of the MIS Manager to participate in project planning at the request of the Project Manager, specifically with respect to the generation of level of effort and schedule estimates. To support the project planning effort, the MIS Manager shall provide a copy of the MIS Request From included as Attachment A to the project manager. It shall be the responsibility of the MIS Manager to generate level of effort and budget estimates at the time database support is requested if a budget does not exist at the time of the request. The MIS Request Form shall be provided to the Project Manager at the time of any such requests. It shall be the responsibility of the MIS Manager to notify the Project Manager of any anticipated level of effort overruns or schedule noncompliances as soon as such problems arise along with full justification for any deviations from the budget estimates (provided they were generated by the MIS Manager). It shall be the responsibility of the MIS Manager to document any changes to the scope of work dictated by the Project Manager, along with an estimate of the impact of the change on the level of effort and the schedule.

Program/Department Managers - It shall be the responsibility of the Department and/or Program Managers (or designees) to inform their respective department's Project Managers of the existence and requirements of this SOP.

Project Manager - It shall be the responsibility of each Project Manager to determine the applicability of this SOP based on: (1) program-specific requirements, and (2) project size and objectives. It shall be the responsibility of the Project Manager (or designee) to ensure that the FOL is familiar with the requirements regarding Chain-of-Custody Form provision to the Database Records Custodian. It shall be the responsibility of the Project Manager (or designee) to determine which, if any, historical data are relevant and to ensure that such data (including all relevant information such as originating entity, sample locations, sampling dates, etc.) are provided to the Database Records Custodian for inclusion in the Database Records File. It shall be the responsibility of the Project Manager to obtain project planning input regarding the level of effort and schedule from the MIS Manager. It shall be the responsibility of the Project Manager to complete the database checklist (Attachment A) to support the level of effort and schedule estimate and to facilitate database preparation and subroutine execution.

Risk Assessment Department Manager - It shall be the responsibility of the Risk Assessment Department Manager to monitor compliance with this Standard Operating Procedure, to modify this SOP as necessary, and to take corrective action if necessary. Monitoring of the process shall be completed on a quarterly basis.

Quality Assurance Reviewers - It shall be the responsibility of the Quality Assurance Reviewers to verify the completeness of the sample results via review of the Chain-of-Custody Forms and Sample Tracking Summaries. It shall be the responsibility of the Quality Assurance Reviewers to ensure the correctness of the database via direct comparison of the hardcopy printout of the database and the hardcopy summaries of the original analytical data (e.g., Form Is provided in data validation deliverables). Correctness includes the presence of all relevant sample information (all sample information fields), agreement of the laboratory and database analytical results, and the presence of data validation qualifiers.

Quality Manager - It shall be the responsibility of the Quality Manager to monitor compliance with this Standard Operating Procedure via routine audits.

5.0 PROCEDURES

5.1 Introduction

Verification of the accuracy and completeness of an electronic database can only be accomplished via comparison of a hardcopy of the database with hardcopy of all relevant sample information. The primary purposes of this SOP are to ensure that 1) all necessary hardcopy information is readily available to Quality Assurance Reviewers; 2) ensure that the Quality Assurance review is completed in a consistent and comprehensive manner, and; 3) ensure that documentation of the Quality Assurance review process is maintained in the project file.

5.2 File Establishment

A Database Record file shall be established for a specific project at the discretion of the Project Manager. Initiation of the filing procedure will commence upon receipt of the first set of Chain-of-Custody documents from a FOL or sampling technician. The Database Record Custodian shall establish a project-specific file for placement in the Database Record File. Each file in the Database Record File shall consist of standard components placed in the file as the project progresses. Each file shall be clearly labeled with the project number, which shall be placed on the front of the file drawer and on each and every hanging file folder relevant to the project. The following constitute the minimum components of a completed file:

- Electronic Deliverables
- Sample Tracking Forms
- Chain-of-Custody Forms
- Data Validation Letters
- Quality Assurance Records

5.3 Electronic Deliverables

The format of electronic deliverables shall be specified in the laboratory procurement specification and shall be provided by the laboratory. The integrity of all original electronic data deliverables shall be maintained. This shall be accomplished via the generation of copies of each electronic deliverable provided by the laboratory. The original electronic deliverable shall be provided to the project manager for inclusion in the project file. A copy of the original electronic deliverable shall be placed in the Database Record File. The second copy shall be maintained by the MIS Manager (or designee) to be used as a working copy.

5.4 Sample Tracking Forms

Updated versions of the sample tracking form for each relevant project shall be maintained by the Database Record Custodian. The Sample Tracking Forms shall be updated any time additional Chain-of-Custody Forms are received from a FOL or sampling technician, or at any time that data are received from a laboratory, or at any time that validation of a given data package (sample delivery group) is completed. The Data Validation Coordinator shall inform the Database Record Custodian of the receipt of any data packages from the laboratory and of completion of validation of a given data package to facilitate updating of the Sample Tracking Form. The Database Record Custodian shall place a revised copy of the Sample Tracking Form in the Database Record File anytime it has been updated. Copies of the updated Sample Tracking Form shall also be provided to the project manager to apprise the project manager of sample package receipt, completion of validation, etc.

5.5 Chain-of-Custody Forms

The Chain-of-Custody Forms for all sampling efforts will be used as the basis for (1) updating the Sample Tracking Form, and (2) confirming that all required samples and associated analyses have been completed. It shall be the responsibility of the FOL (or sample technician) to provide a photocopy of all Chain-of-Custody Forms to the Database Record Custodian immediately upon completion of a sampling effort. The Database Record Custodian shall then place the copies of the Chain-of-Custody Form(s) in the Database Record File. Upon receipt of a sample data package from an analytical laboratory, the Data Validation Coordinator shall provide a copy of the laboratory Chain-of-Custody Form to the Database Record Custodian. The Database Record Custodian shall use this copy to update the Sample Tracking Summary and shall place the copy of the laboratory-provided Chain-of-Custody Form in the Database Record File. The photocopy of the laboratory-provided Chain-of Custody Form shall be stapled to the previously filed field copy. Upon receipt of all analytical data, two copies of the Chain-of-Custody will therefore be in the file. Review of the Chain-of-Custody Forms will therefore be a simple mechanism to determine if all data have been received. Chain-of-Custody is addressed in SOP SA-6.1.

5.6 Data Validation Letters

All data validation deliverables (or raw data summaries if validation is not conducted) shall be provided for inclusion in both the Database Record File and the project file. If USEPA regional- or client-specific requirements are such that Form Is (or similar analytical results) need not be provided with the validation deliverable, copies of such results must be appended to the deliverable. It is preferable, although not essential that the validation qualifiers be hand-written directly on the data summary forms. The data validation deliverables (and attendant analytical summaries) will provide the basis for direct comparison of the database printout and the raw data and qualifiers.

5.7 Historical Data

At the direction of the Project Manager, historical data may also be included in a project-specific analytical database. In the event that historical data are germane to the project, hardcopy of the historical data must be included in the Database Record File. Historical data may be maintained in the form of final reports or as raw data. The information contained in the historical data file must be sufficient to identify its origin, its collection date, the sample location, the matrix, and any and all other pertinent information. All available analytical data, Chain-of-Custody Forms, boring logs, well construction logs, sample location maps, shall be photocopied by the Project Manager (or designee) and placed in one or more 3-ring binders. All information shall be organized chronologically by matrix. It shall be the responsibility of the Project Manager (or designee) to ensure that all inconsistencies between analytical data, Chain-of-Custody Forms, boring logs, sample log sheets, and field logbooks are identified and corrected. The Project Manager (or designee) shall decide which nomenclature is appropriate and edit, initial and date all relevant forms. Data entry may only be performed on information that has undergone the aforementioned

editing process, thereby having a direct correlation between hardcopy information and what will become the electronic database.

6.0 RECORDS

Records regarding database preparation and quality assurance review include all those identified in the previous section. Upon completion of the database task, records from the file will be forwarded to the Project Manager for inclusion in the project file, or will be placed in bankers boxes (or equivalent) for storage. The final records for storage shall include the following minimum information on placards placed on both the top and end of the storage box:

Database Record File
PROJECT NUMBER: ____
SITE NAME: _____
DATE FILED: __/__/__
SUMMARY OF CONTENTS ENCLOSED
BOX _ OF _

Project- or program-specific record keeping requirements shall take precedence over the record keeping requirements of this SOP.

ATTACHMENT A

MIS REQUEST FORM

Tetra Tech NUS, Inc.

Project Name: | Request Date:
CTO: | Date Data Available for Production:
Project Manager: | Request in Support of:
Requestor: | Database Lead:
Program/Client: | GIS Lead:
State/EPA Region: | Statistics Lead:
| Risk Lead:
Site Name(s) (Area, OU, etc.):
Sampling Date(s):
Matrix: ☐ GW ☐ SO ☐ SD ☐ SW ☐ Other:

Labels: ☐ Labels needed for an upcoming sampling event _____ Total # of Samples
_____ Estimated Hours | Additional Instructions:
_____ Due Date
_____ Complete ETS Charge No.
_____ FOL

Data Entry:
☐ Chemical data needs to be entered from hardcopy _____ Estimated # of Samples
☐ Chemical data needs to be formated electronically
☐ Field analytical data needs to be entered from hardcopy
☐ Geologic data needs to be entered from hardcopy
☐ Hydrology data needs to be entered from hardcopy
_____ Estimated Hours | Additional Instructions:
_____ Due Date
_____ Complete ETS Charge No.

Tables:
☐ Full Data Printout
☐ Summary of Positive Hits
☐ Occurance and Distribution ☐ with criteria
☐ Sampling Analytical Summary
☐ Other:
_____ Estimated Hours | Additional Instructions:
_____ Due Date
_____ Complete ETS Charge No.

GIS:
☐ General Facility Location
☐ Site Location
☐ Potentiometric Contours/Groundwater Flow
☐ Sample Location Proposed
☐ Sample Location Existing
☐ Tag Map Single Round
☐ Tag Map Multiple Round
☐ Isoconcentrations
☐ Chart Map
☐ 3D Visualization
☐ EGIS CD
☐ Other:
_____ Estimated Hours | Additional Instructions:
_____ Due Date
_____ Complete ETS Charge No.

Statistics: ☐ Yes
_____ Estimated Hours | Additional Instructions:
_____ Due Date
_____ Complete ETS Charge No.

Geostatistics: ☐ Yes
_____ Estimated Hours | Additional Instructions:
_____ Due Date
_____ Complete ETS Charge No.

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION I

LOW STRESS (low flow) PURGING AND SAMPLING PROCEDURE FOR THE COLLECTION OF GROUNDWATER SAMPLES FROM MONITORING WELLS

Quality Assurance Unit
U.S. Environmental Protection Agency – Region 1
11 Technology Drive
North Chelmsford, MA 01863

Prepared by: Charles Porfert 1/19/10
(Charles Porfert, Quality Assurance Unit) Date

Approved by: Gerard Sotolongo 1-19-10
(Gerard Sotolongo, Quality Assurance Unit) Date

Revision Page

Date	Rev #	Summary of changes	Sections
7/30/96	2	Finalized	
01/19/10	3	Updated	All sections

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USE OF TERMS

Equipment blank: The equipment blank shall include the pump and the pump's tubing. If tubing is dedicated to the well, the equipment blank needs only to include the pump in subsequent sampling rounds. If the pump and tubing are dedicated to the well, the equipment blank is collected prior to its placement in the well. If the pump and tubing will be used to sample multiple wells, the equipment blank is normally collected after sampling from contaminated wells and not after background wells.

Field duplicates: Field duplicates are collected to determine precision of the sampling procedure. For this procedure, collect duplicate for each analyte group in consecutive order (VOC original, VOC duplicate, SVOC original, SVOC duplicate, etc.).

Indicator field parameters: This SOP uses field measurements of turbidity, dissolved oxygen, specific conductance, temperature, pH, and oxidation/reduction potential (ORP) as indicators of when purging operations are sufficient and sample collection may begin.

Matrix Spike/Matrix Spike Duplicates: Used by the laboratory in its quality assurance program. Consult the laboratory for the sample volume to be collected.

Poteniometric Surface: The level to which water rises in a tightly cased well constructed in a confined aquifer. In an unconfined aquifer, the potentiometric surface is the water table.

QAPP: Quality Assurance Project Plan

SAP: Sampling and Analysis Plan

SOP: Standard operating procedure

Stabilization: A condition that is achieved when all indicator field parameter measurements are sufficiently stable (as described in the "Monitoring Indicator Field Parameters" section) to allow sample collection to begin.

Temperature blank: A temperature blank is added to each sample cooler. The blank is measured upon receipt at the laboratory to assess whether the samples were properly cooled during transit.

Trip blank (VOCs): Trip blank is a sample of analyte-free water taken to the sampling site and returned to the laboratory. The trip blanks (one pair) are added to each sample cooler that contains VOC samples.

SCOPE & APPLICATION

The goal of this groundwater sampling procedure is to collect water samples that reflect the total mobile organic and inorganic loads (dissolved and colloidal sized fractions) transported through the subsurface under ambient flow conditions, with minimal physical and chemical alterations from sampling operations. This standard operating procedure (SOP) for collecting groundwater samples will help ensure that the project's data quality objectives (DQOs) are met under certain low-flow conditions.

The SOP emphasizes the need to minimize hydraulic stress at the well-aquifer interface by maintaining low water-level drawdowns, and by using low pumping rates during purging and sampling operations. Indicator field parameters (e.g., dissolved oxygen, pH, etc.) are monitored during purging in order to determine when sample collection may begin. Samples properly collected using this SOP are suitable for analysis of groundwater contaminants (volatile and semi-volatile organic analytes, dissolved gases, pesticides, PCBs, metals and other inorganics), or naturally occurring analytes. This SOP is based on Puls, and Barcelona (1996).

This procedure is designed for monitoring wells with an inside diameter (1.5-inches or greater) that can accommodate a positive lift pump with a screen length or open interval ten feet or less and with a water level above the top of the screen or open interval (Hereafter, the "screen or open interval" will be referred to only as "screen interval"). This SOP is not applicable to other well-sampling conditions.

While the use of dedicated sampling equipment is not mandatory, dedicated pumps and tubing can reduce sampling costs significantly by streamlining sampling activities and thereby reducing the overall field costs.

The goal of this procedure is to emphasize the need for consistency in deploying and operating equipment while purging and sampling monitoring wells during each sampling event. This will help to minimize sampling variability.

This procedure describes a general framework for groundwater sampling. Other site specific information (hydrogeological context, conceptual site model (CSM), DQOs, etc.) coupled with systematic planning must be added to the procedure in order to develop an appropriate site specific SAP/QAPP. In addition, the site specific SAP/QAPP must identify the specific equipment that will be used to collect the groundwater samples.

This procedure does not address the collection of water or free product samples from wells containing free phase LNAPLs and/or DNAPLs (light or dense non-aqueous phase

liquids). For this type of situation, the reader may wish to check: Cohen, and Mercer (1993) or other pertinent documents.

This SOP is to be used when collecting groundwater samples from monitoring wells at all Superfund, Federal Facility and RCRA sites in Region 1 under the conditions described herein. Request for modification of this SOP, in order to better address specific situations at individual wells, must include adequate technical justification for proposed changes. All changes and modifications must be approved and included in a revised SAP/QAPP before implementation in field.

BACKGROUND FOR IMPLEMENTATION

It is expected that the monitoring well screen has been properly located (both laterally and vertically) to intercept existing contaminant plume(s) or along flow paths of potential contaminant migration. Problems with inappropriate monitoring well placement or faulty/improper well installation cannot be overcome by even the best water sampling procedures. This SOP presumes that the analytes of interest are moving (or will potentially move) primarily through the more permeable zones intercepted by the screen interval.

Proper well construction, development, and operation and maintenance cannot be overemphasized. The use of installation techniques that are appropriate to the hydrogeologic setting of the site often prevent "problem well" situations from occurring. During well development, or redevelopment, tests should be conducted to determine the hydraulic characteristics of the monitoring well. The data can then be used to set the purging/sampling rate, and provide a baseline for evaluating changes in well performance and the potential need for well rehabilitation. Note: if this installation data or well history (construction and sampling) is not available or discoverable, for all wells to be sampled, efforts to build a sampling history should commence with the next sampling event.

The pump intake should be located within the screen interval and at a depth that will remain under water at all times. It is recommended that the intake depth and pumping rate remain the same for all sampling events. The mid-point or the lowest historical midpoint of the saturated screen length is often used as the location of the pump intake. For new wells, or for wells without pump intake depth information, the site's SAP/QAPP must provide clear reasons and instructions on how the pump intake depth(s) will be selected, and reason(s) for the depth(s) selected. If the depths to top and bottom of the well screen are not known, the SAP/QAPP will need to describe how the sampling depth will be determined and how the data can be used.

Stabilization of indicator field parameters is used to indicate that conditions are suitable for sampling to begin. Achievement of turbidity levels of less than 5 NTU, and stable drawdowns of less than 0.3 feet, while desirable, are not mandatory. Sample collection

may still take place provided the indicator field parameter criteria in this procedure are met. If after 2 hours of purging indicator field parameters have not stabilized, one of three optional courses of action may be taken: a) continue purging until stabilization is achieved, b) discontinue purging, do not collect any samples, and record in log book that stabilization could not be achieved (documentation must describe attempts to achieve stabilization), c) discontinue purging, collect samples and provide full explanation of attempts to achieve stabilization (note: there is a risk that the analytical data obtained, especially metals and strongly hydrophobic organic analytes, may reflect a sampling bias and therefore, the data may not meet the data quality objectives of the sampling event).

It is recommended that low-flow sampling be conducted when the air temperature is above 32°F (0°C). If the procedure is used below 32°F, special precautions will need to be taken to prevent the groundwater from freezing in the equipment. Because sampling during freezing temperatures may adversely impact the data quality objectives, the need for water sample collection during months when these conditions are likely to occur should be evaluated during site planning and special sampling measures may need to be developed. Ice formation in the flow-through-cell will cause the monitoring probes to act erratically. A transparent flow-through-cell needs to be used to observe if ice is forming in the cell. If ice starts to form on the other pieces of the sampling equipment, additional problems may occur.

HEALTH & SAFETY

When working on-site, comply with all applicable OSHA requirements and the site's health/safety procedures. All proper personal protection clothing and equipment are to be worn. Some samples may contain biological and chemical hazards. These samples should be handled with suitable protection to skin, eyes, etc.

CAUTIONS

The following cautions need to be considered when planning to collect groundwater samples when the below conditions occur.

If the groundwater degasses during purging of the monitoring well, dissolved gases and VOCs will be lost. When this happens, the groundwater data for dissolved gases (e.g., methane, ethene, ethane, dissolved oxygen, etc.) and VOCs will need to be qualified. Some conditions that can promote degassing are the use of a vacuum pump (e.g., peristaltic pumps), changes in aperture along the sampling tubing, and squeezing/pinching the pump's tubing which results in a pressure change.

When collecting the samples for dissolved gases and VOCs analyses, avoid aerating the groundwater in the pump's tubing. This can cause loss of the dissolved gases and VOCs in

the groundwater. Having the pump's tubing completely filled prior to sampling will avoid this problem when using a centrifugal pump or peristaltic pump.

Direct sun light and hot ambient air temperatures may cause the groundwater in the tubing and flow-through-cell to heat up. This may cause the groundwater to degas which will result in loss of VOCs and dissolved gases. When sampling under these conditions, the sampler will need to shade the equipment from the sunlight (e.g., umbrella, tent, etc.). If possible, sampling on hot days, or during the hottest time of the day, should be avoided. The tubing exiting the monitoring well should be kept as short as possible to avoid the sun light or ambient air from heating up the groundwater.

Thermal currents in the monitoring well may cause vertical mixing of water in the well bore. When the air temperature is colder than the groundwater temperature, it can cool the top of the water column. Colder water which is denser than warm water sinks to the bottom of the well and the warmer water at the bottom of the well rises, setting up a convention cell. "During low-flow sampling, the pumped water may be a mixture of convecting water from within the well casing and aquifer water moving inward through the screen. This mixing of water during low-flow sampling can substantially increase equilibration times, can cause false stabilization of indicator parameters, can give false indication of redox state, and can provide biological data that are not representative of the aquifer conditions" (Vroblesky 2007).

Failure to calibrate or perform proper maintenance on the sampling equipment and measurement instruments (e.g., dissolved oxygen meter, etc.) can result in faulty data being collected.

Interferences may result from using contaminated equipment, cleaning materials, sample containers, or uncontrolled ambient/surrounding air conditions (e.g., truck/vehicle exhaust nearby).

Cross contamination problems can be eliminated or minimized through the use of dedicated sampling equipment and/or proper planning to avoid ambient air interferences. Note that the use of dedicated sampling equipment can also significantly reduce the time needed to complete each sampling event, will promote consistency in the sampling, and may reduce sampling bias by having the pump's intake at a constant depth.

Clean and decontaminate all sampling equipment prior to use. All sampling equipment needs to be routinely checked to be free from contaminants and equipment blanks collected to ensure that the equipment is free of contaminants. Check the previous equipment blank data for the site (if they exist) to determine if the previous cleaning procedure removed the contaminants. If contaminants were detected and they are a concern, then a more vigorous cleaning procedure will be needed.

PERSONNEL QUALIFICATIONS

All field samplers working at sites containing hazardous waste must meet the requirements of the OSHA regulations. OSHA regulations may require the sampler to take the 40 hour OSHA health and safety training course and a refresher course prior to engaging in any field activities, depending upon the site and field conditions.

The field samplers must be trained prior to the use of the sampling equipment, field instruments, and procedures. Training is to be conducted by an experienced sampler before initiating any sampling procedure.

The entire sampling team needs to read, and be familiar with, the site Health and Safety Plan, all relevant SOPs, and SAP/QAPP (and the most recent amendments) before going onsite for the sampling event. It is recommended that the field sampling leader attest to the understanding of these site documents and that it is recorded.

EQUIPMENT AND SUPPLIES

A. Informational materials for sampling event

A copy of the current Health and Safety Plan, SAP/QAPP, monitoring well construction data, location map(s), field data from last sampling event, manuals for sampling, and the monitoring instruments' operation, maintenance, and calibration manuals should be brought to the site.

B. Well keys.

C. Extraction device

Adjustable rate, submersible pumps (e.g., centrifugal, bladder, etc.) which are constructed of stainless steel or Teflon are preferred. Note: if extraction devices constructed of other materials are to be used, adequate information must be provided to show that the substituted materials do not leach contaminants nor cause interferences to the analytical procedures to be used. Acceptance of these materials must be obtained before the sampling event.

If bladder pumps are selected for the collection of VOCs and dissolved gases, the pump setting should be set so that one pulse will deliver a water volume that is sufficient to fill a 40 mL VOC vial. This is not mandatory, but is considered a "best practice". For the proper operation, the bladder pump will need a minimum amount of water above the pump; consult the manufacturer for the recommended submergence. The pump's recommended submergence value should be determined during the planning stage, since it may influence well construction and placement of dedicated pumps where water-level fluctuations are significant.

Adjustable rate, peristaltic pumps (suction) are to be used with caution when collecting samples for VOCs and dissolved gases (e.g., methane, carbon dioxide, etc.) analyses. Additional information on the use of peristaltic pumps can be found in Appendix A. If peristaltic pumps are used, the inside diameter of the rotor head tubing needs to match the inside diameter of the tubing installed in the monitoring well.

Inertial pumping devices (motor driven or manual) are not recommended. These devices frequently cause greater disturbance during purging and sampling, and are less easily controlled than submersible pumps (potentially increasing turbidity and sampling variability, etc.). This can lead to sampling results that are adversely affected by purging and sampling operations, and a higher degree of data variability.

D. Tubing

Teflon or Teflon-lined polyethylene tubing are preferred when sampling is to include VOCs, SVOCs, pesticides, PCBs and inorganics. Note: if tubing constructed of other materials is to be used, adequate information must be provided to show that the substituted materials do not leach contaminants nor cause interferences to the analytical procedures to be used. Acceptance of these materials must be obtained before the sampling event.

PVC, polypropylene or polyethylene tubing may be used when collecting samples for metal and other inorganics analyses.

The use of 1/4 inch or 3/8 inch (inside diameter) tubing is recommended. This will help ensure that the tubing remains liquid filled when operating at very low pumping rates when using centrifugal and peristaltic pumps.

Silastic tubing should be used for the section around the rotor head of a peristaltic pump. It should be less than a foot in length. The inside diameter of the tubing used at the pump rotor head must be the same as the inside diameter of tubing placed in the well. A tubing connector is used to connect the pump rotor head tubing to the well tubing. Alternatively, the two pieces of tubing can be connected to each other by placing the one end of the tubing inside the end of the other tubing. The tubing must not be reused.

E. The water level measuring device

Electronic "tape", pressure transducer, water level sounder/level indicator, etc. should be capable of measuring to 0.01 foot accuracy. Recording pressure transducers, mounted above the pump, are especially helpful in tracking water levels during pumping operations, but their use must include check measurements with a water level "tape" at the start and end of each sampling event.

F. Flow measurement supplies

Graduated cylinder (size according to flow rate) and stopwatch usually will suffice.

Large graduated bucket used to record total water purged from the well.

G. Interface probe

To be used to check on the presence of free phase liquids (LNAPL, or DNAPL) before purging begins (as needed).

H. Power source (generator, nitrogen tank, battery, etc.)

When a gasoline generator is used, locate it downwind and at least 30 feet from the well so that the exhaust fumes do not contaminate samples.

I. Indicator field parameter monitoring instruments

Use of a multi-parameter instrument capable of measuring pH, oxidation/reduction potential (ORP), dissolved oxygen (DO), specific conductance, temperature, and coupled with a flow-through-cell is required when measuring all indicator field parameters, except turbidity. Turbidity is collected using a separate instrument. Record equipment/instrument identification (manufacturer, and model number).

Transparent, small volume flow-through-cells (e.g., 250 mLs or less) are preferred. This allows observation of air bubbles and sediment buildup in the cell, which can interfere with the operation of the monitoring instrument probes, to be easily detected. A small volume cell facilitates rapid turnover of water in the cell between measurements of the indicator field parameters.

It is recommended to use a flow-through-cell and monitoring probes from the same manufacturer and model to avoid incompatibility between the probes and flow-through-cell.

Turbidity samples are collected before the flow-through-cell. A "T" connector coupled with a valve is connected between the pump's tubing and flow-through-cell. When a turbidity measurement is required, the valve is opened to allow the groundwater to flow into a container. The valve is closed and the container sample is then placed in the turbidimeter.

Standards are necessary to perform field calibration of instruments. A minimum of two standards are needed to bracket the instrument measurement range for all parameters except ORP which use a Zobell solution as a standard. For dissolved oxygen, a wet sponge used for the 100% saturation and a zero dissolved oxygen solution are used for the calibration.

Barometer (used in the calibration of the Dissolved Oxygen probe) and the conversion formula to convert the barometric pressure into the units of measure used by the Dissolved Oxygen meter are needed.

J. Decontamination supplies

Includes (for example) non-phosphate detergent, distilled/deionized water, isopropyl alcohol, etc.

K. Record keeping supplies

Logbook(s), well purging forms, chain-of-custody forms, field instrument calibration forms, etc.

L. Sample bottles

M. Sample preservation supplies (as required by the analytical methods)

N. Sample tags or labels

O. PID or FID instrument

If appropriate, to detect VOCs for health and safety purposes, and provide qualitative field evaluations.

P. Miscellaneous Equipment

Equipment to keep the sampling apparatus shaded in the summer (e.g., umbrella) and from freezing in the winter. If the pump's tubing is allowed to heat up in the warm weather, the cold groundwater may degas as it is warmed in the tubing.

EQUIPMENT/INSTRUMENT CALIBRATION

Prior to the sampling event, perform maintenance checks on the equipment and instruments according to the manufacturer's manual and/or applicable SOP. This will ensure that the equipment/instruments are working properly before they are used in the field.

Prior to sampling, the monitoring instruments must be calibrated and the calibration documented. The instruments are calibrated using U.S Environmental Protection Agency Region 1 *Calibration of Field Instruments (temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction [ORP], and turbidity),* January 19, 2010, or latest version or from one of the methods listed in 40CFR136, 40CFR141 and SW-846.

The instruments shall be calibrated at the beginning of each day. If the field measurement falls outside the calibration range, the instrument must be re-calibrated so that all measurements fall within the calibration range. At the end of each day, a calibration check is performed to verify that instruments remained in calibration throughout the day. This check is performed while the instrument is in measurement mode, not calibration mode. If the field instruments are being used to monitor the natural attenuation parameters, then a calibration check at mid-day is highly recommended to ensure that the instruments did not drift out of calibration. Note: during the day if the instrument reads zero or a negative number for dissolved oxygen, pH, specific conductance, or turbidity (negative value only), this indicates that the instrument drifted out of calibration or the instrument is malfunctioning. If this situation occurs the data from this instrument will need to be qualified or rejected.

PRELIMINARY SITE ACTIVITIES (as applicable)

Check the well for security (damage, evidence of tampering, missing lock, etc.) and record pertinent observations (include photograph as warranted).

If needed lay out sheet of clean polyethylene for monitoring and sampling equipment, unless equipment is elevated above the ground (e.g., on a table, etc.).

Remove well cap and if appropriate measure VOCs at the rim of the well with a PID or FID instrument and record reading in field logbook or on the well purge form.

If the well casing does not have an established reference point (usually a V-cut or indelible mark in the well casing), make one. Describe its location and record the date of the mark in the logbook (consider a photographic record as well). All water level measurements must be recorded relative to this reference point (and the altitude of this point should be determined using techniques that are appropriate to site's DQOs.

If water-table or potentiometric surface map(s) are to be constructed for the sampling event, perform synoptic water level measurement round (in the shortest possible time) before any purging and sampling activities begin. If possible, measure water level depth (to 0.01 ft.) and total well depth (to 0.1 ft.) the day before sampling begins, in order to allow for re-settlement of any particulates in the water column. This is especially important for those wells that have not been recently sampled because sediment buildup in the well may require the well to be redeveloped. If measurement of total well depth is not made the day before, it should be measured after sampling of the well is complete. All measurements must be taken from the established referenced point. Care should be taken to minimize water column disturbance.

Check newly constructed wells for the presence of LNAPLs or DNAPLs before the initial sampling round. If none are encountered, subsequent check measurements with an interface probe may not be necessary unless analytical data or field analysis signal a worsening situation. This SOP cannot be used in the presence of LNAPLs or DNAPLs. If NAPLs are present, the project team must decide upon an alternate sampling method. All project modifications must be approved and documented prior to implementation.

If available check intake depth and drawdown information from previous sampling event(s) for each well. Duplicate, to the extent practicable, the intake depth and extraction rate (use final pump dial setting information) from previous event(s). If changes are made in the intake depth or extraction rate(s) used during previous sampling event(s), for either portable or dedicated extraction devices, record new values, and explain reasons for the changes in the field logbook.

PURGING AND SAMPLING PROCEDURE

Purging and sampling wells in order of increasing chemical concentrations (known or anticipated) are preferred.

The use of dedicated pumps is recommended to minimize artificial mobilization and entrainment of particulates each time the well is sampled. Note that the use of dedicated sampling equipment can also significantly reduce the time needed to complete each

sampling event, will promote consistency in the sampling, and may reduce sampling bias by having the pump's intake at a constant depth.

A. Initial Water Level

Measure the water level in the well before installing the pump if a non-dedicated pump is being used. The initial water level is recorded on the purge form or in the field logbook.

B. Install Pump

Lower pump, safety cable, tubing and electrical lines slowly (to minimize disturbance) into the well to the appropriate depth (may not be the mid-point of the screen/open interval). The Sampling and Analysis Plan/Quality Assurance Project Plan should specify the sampling depth (used previously), or provide criteria for selection of intake depth for each new well. If possible keep the pump intake at least two feet above the bottom of the well, to minimize mobilization of particulates present in the bottom of the well.

Pump tubing lengths, above the top of well casing should be kept as short as possible to minimize heating the groundwater in the tubing by exposure to sun light and ambient air temperatures. Heating may cause the groundwater to degas, which is unacceptable for the collection of samples for VOC and dissolved gases analyses.

C. Measure Water Level

Before starting pump, measure water level. Install recording pressure transducer, if used to track drawdowns, to initialize starting condition.

D. Purge Well

From the time the pump starts purging and until the time the samples are collected, the purged water is discharged into a graduated bucket to determine the total volume of groundwater purged. This information is recorded on the purge form or in the field logbook.

Start the pump at low speed and slowly increase the speed until discharge occurs. Check water level. Check equipment for water leaks and if present fix or replace the affected equipment. Try to match pumping rate used during previous sampling event(s). Otherwise, adjust pump speed until there is little or no water level drawdown. If the minimal drawdown that can be achieved exceeds 0.3 feet, but remains stable, continue purging.

Monitor and record the water level and pumping rate every five minutes (or as appropriate) during purging. Record any pumping rate adjustments (both time and flow rate). Pumping rates should, as needed, be reduced to the minimum capabilities of the pump to ensure stabilization of the water level. Adjustments are best made in the first fifteen minutes of pumping in order to help minimize purging time. During pump start-up, drawdown may exceed the 0.3 feet target and then "recover" somewhat as pump flow adjustments are made. Purge volume calculations should utilize stabilized drawdown value, not the initial drawdown. If the initial water level is above the top of the screen do not allow the water level to fall into the well screen. The final purge volume must be greater than the stabilized drawdown volume plus the pump's tubing volume. If the drawdown has exceeded 0.3 feet and stabilizes, calculate the volume of water between the initial water level and the stabilized water level. Add the volume of the water which occupies the pump's tubing to this calculation. This combined volume of water needs to be purged from the well after the water level has stabilized before samples are collected.

Avoid the use of constriction devices on the tubing to decrease the flow rate because the constrictor will cause a pressure difference in the water column. This will cause the groundwater to degas and result in a loss of VOCs and dissolved gasses in the groundwater samples.

Note: the flow rate used to achieve a stable pumping level should remain constant while monitoring the indicator parameters for stabilization and while collecting the samples.

Wells with low recharge rates may require the use of special pumps capable of attaining very low pumping rates (e.g., bladder, peristaltic), and/or the use of dedicated equipment. For new monitoring wells, or wells where the following situation has not occurred before, if the recovery rate to the well is less than 50 mL/min., or the well is being essentially dewatered during purging, the well should be sampled as soon as the water level has recovered sufficiently to collect the volume needed for all anticipated samples. The project manager or field team leader will need to make the decision when samples should be collected, how the sample is to be collected, and the reasons recorded on the purge form or in the field logbook. A water level measurement needs to be performed and recorded before samples are collected. If the project manager decides to collect the samples using the pump, it is best during this recovery period that the pump intake tubing not be removed, since this will aggravate any turbidity problems. Samples in this specific situation may be collected without stabilization of indicator field parameters. Note that field conditions and efforts to overcome problematic situations must be recorded in order to support field decisions to deviate from normal procedures described in this SOP. If this type of problematic situation persists in a well, then water sample collection should be changed to a passive or no-purge method, if consistent with the site's DQOs, or have a new well installed.

E. Monitor Indicator Field Parameters

After the water level has stabilized, connect the "T" connector with a valve and the flow-through-cell to monitor the indicator field parameters. If excessive turbidity is anticipated or encountered with the pump startup, the well may be purged for a while without connecting up the flow-through-cell, in order to minimize particulate buildup in the cell (This is a judgment call made by the sampler). Water level drawdown measurements should be made as usual. If possible, the pump may be installed the day before purging to allow particulates that were disturbed during pump insertion to settle.

During well purging, monitor indicator field parameters (turbidity, temperature, specific conductance, pH, ORP, DO) at a frequency of five minute intervals or greater. The pump's flow rate must be able to "turn over" at least one flow-through-cell volume between measurements (for a 250 mL flow-through-cell with a flow rate of 50 mLs/min., the monitoring frequency would be every five minutes; for a 500 mL flow-through-cell it would be every ten minutes). If the cell volume cannot be replaced in the five minute interval, then the time between measurements must be increased accordingly. Note: during the early phase of purging emphasis should be put on minimizing and stabilizing pumping stress, and recording those adjustments followed by stabilization of indicator parameters. Purging is considered complete and sampling may begin when all the above indicator field parameters have stabilized. Stabilization is considered to be achieved when three consecutive readings are within the following limits:

Turbidity (10% for values greater than 5 NTU; if three Turbidity values are less than 5 NTU, consider the values as stabilized),
Dissolved Oxygen (10% for values greater than 0.5 mg/L, if three Dissolved Oxygen values are less than 0.5 mg/L, consider the values as stabilized),
Specific Conductance (3%),
Temperature (3%),
pH (± 0.1 unit),
Oxidation/Reduction Potential (±10 millivolts).

All measurements, except turbidity, must be obtained using a flow-through-cell. Samples for turbidity measurements are obtained before water enters the flow-through-cell. Transparent flow-through-cells are preferred, because they allow field personnel to watch for particulate build-up within the cell. This build-up may affect indicator field parameter values measured within the cell. If the cell needs to be cleaned during purging operations, continue pumping and disconnect cell for cleaning, then reconnect after cleaning and continue monitoring activities. Record start and stop times and give a brief description of cleaning activities.

The flow-through-cell must be designed in a way that prevents gas bubble entrapment in the cell. Placing the flow-through-cell at a 45 degree angle with the port facing upward can help remove bubbles from the flow-through-cell (see Appendix B Low-Flow Setup Diagram). All during the measurement process, the flow-through-cell must remain free of any gas bubbles. Otherwise, the monitoring probes may act erratically. When the pump is turned off or cycling on/off (when using a bladder pump), water in the cell must not drain out. Monitoring probes must remain submerged in water at all times.

F. Collect Water Samples

When samples are collected for laboratory analyses, the pump's tubing is disconnected from the "T" connector with a valve and the flow-through-cell. The samples are collected directly from the pump's tubing. Samples must not be collected from the flow-through-cell or from the "T" connector with a valve.

VOC samples are normally collected first and directly into pre-preserved sample containers. However, this may not be the case for all sampling locations; the SAP/QAPP should list the order in which the samples are to be collected based on the project's objective(s). Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

If the pump's flow rate is too high to collect the VOC/dissolved gases samples, collect the other samples first. Lower the pump's flow rate to a reasonable rate and collect the VOC/dissolved gases samples and record the new flow rate.

During purging and sampling, the centrifugal/peristaltic pump tubing must remain filled with water to avoid aeration of the groundwater. It is recommended that 1/4 inch or 3/8 inch (inside diameter) tubing be used to help insure that the sample tubing remains water filled. If the pump tubing is not completely filled to the sampling point, use the following procedure to collect samples: collect non-VOC/dissolved gases samples first, then increase flow rate slightly until the water completely fills the tubing, collect the VOC/dissolved gases samples, and record new drawdown depth and flow rate.

For bladder pumps that will be used to collect VOC or dissolved gas samples, it is recommended that the pump be set to deliver long pulses of water so that one pulse will fill a 40 mL VOC vial.

Use pre-preserved sample containers or add preservative, as required by analytical methods, to the samples immediately after they are collected. Check the analytical methods (e.g. EPA SW-846, 40 CFR 136, water supply, etc.) for additional information on preservation.

If determination of filtered metal concentrations is a sampling objective, collect filtered water samples using the same low flow procedures. The use of an in-line filter (transparent housing preferred) is required, and the filter size (0.45 μm is commonly used) should be based on the sampling objective. Pre-rinse the filter with groundwater prior to sample collection. Make sure the filter is free of air bubbles before samples are collected. Preserve the filtered water sample immediately. Note: filtered water samples are not an acceptable substitute for unfiltered samples when the monitoring objective is to obtain chemical concentrations of total mobile contaminants in groundwater for human health or ecological risk calculations.

Label each sample as collected. Samples requiring cooling will be placed into a cooler with ice or refrigerant for delivery to the laboratory. Metal samples after acidification to a pH less than 2 do not need to be cooled.

G. Post Sampling Activities

If a recording pressure transducer is used to track drawdown, re-measure water level with tape.

After collection of samples, the pump tubing may be dedicated to the well for re-sampling (by hanging the tubing inside the well), decontaminated, or properly discarded.

Before securing the well, measure and record the well depth (to 0.1 ft.), if not measured the day before purging began. Note: measurement of total well depth annually is usually sufficient after the initial low stress sampling event. However, a greater frequency may be needed if the well has a "silting" problem or if confirmation of well identity is needed.

Secure the well.

DECONTAMINATION

Decontaminate sampling equipment prior to use in the first well and then following sampling of each well. Pumps should not be removed between purging and sampling operations. The pump, tubing, support cable and electrical wires which were in contact with the well should be decontaminated by one of the procedures listed below.

The use of dedicated pumps and tubing will reduce the amount of time spent on decontamination of the equipment. If dedicated pumps and tubing are used, only the initial sampling event will require decontamination of the pump and tubing.

Note if the previous equipment blank data showed that contaminant(s) were present after using the below procedure or the one described in the SAP/QAPP, a more vigorous procedure may be needed.

Procedure 1

Decontaminating solutions can be pumped from either buckets or short PVC casing sections through the pump and tubing. The pump may be disassembled and flushed with the decontaminating solutions. It is recommended that detergent and alcohol be used sparingly in the decontamination process and water flushing steps be extended to ensure that any sediment trapped in the pump is removed. The pump exterior and electrical wires must be rinsed with the decontaminating solutions, as well. The procedure is as follows:

Flush the equipment/pump with potable water.

Flush with non-phosphate detergent solution. If the solution is recycled, the solution must be changed periodically.

Flush with potable or distilled/deionized water to remove all of the detergent solution. If the water is recycled, the water must be changed periodically.

Optional - flush with isopropyl alcohol (pesticide grade; must be free of ketones {e.g., acetone}) or with methanol. This step may be required if the well is highly contaminated or if the equipment blank data from the previous sampling event show that the level of contaminants is significant.

Flush with distilled/deionized water. This step must remove all traces of alcohol (if used) from the equipment. The final water rinse must not be recycled.

Procedure 2

Steam clean the outside of the submersible pump.

Pump hot potable water from the steam cleaner through the inside of the pump. This can be accomplished by placing the pump inside a three or four inch diameter PVC pipe with end cap. Hot water from the steam cleaner jet will be directed inside the PVC pipe and the pump exterior will be cleaned. The hot water from the steam cleaner will then be pumped from the PVC pipe through the pump and collected into another container. Note: additives or solutions should not be added to the steam cleaner.

Pump non-phosphate detergent solution through the inside of the pump. If the solution is recycled, the solution must be changed periodically.

Pump potable water through the inside of the pump to remove all of the detergent solution. If the solution is recycled, the solution must be changed periodically.

Pump distilled/deionized water through the pump. The final water rinse must not be recycled.

FIELD QUALITY CONTROL

Quality control samples are required to verify that the sample collection and handling process has not compromised the quality of the groundwater samples. All field quality control samples must be prepared the same as regular investigation samples with regard to sample volume, containers, and preservation. Quality control samples include field duplicates, equipment blanks, matrix spike/matrix spike duplicates, trip blanks (VOCs), and temperature blanks.

FIELD LOGBOOK

A field log shall be kept to document all groundwater field monitoring activities (see Appendix C, example table), and record the following for each well:

Site name, municipality, state.

Well identifier, latitude-longitude or state grid coordinates.

Measuring point description (e.g., north side of PVC pipe).

Well depth, and measurement technique.

Well screen length.

Pump depth.

Static water level depth, date, time and measurement technique.

Presence and thickness of immiscible liquid (NAPL) layers and detection method.

Pumping rate, drawdown, indicator parameters values, calculated or measured total volume pumped, and clock time of each set of measurements.

Type of tubing used and its length.

Type of pump used.

Clock time of start and end of purging and sampling activity.

Types of sample bottles used and sample identification numbers.

Preservatives used.

Parameters requested for analyses.

Field observations during sampling event.

Name of sample collector(s).

Weather conditions, including approximate ambient air temperature.

QA/QC data for field instruments.

Any problems encountered should be highlighted.

Description of all sampling/monitoring equipment used, including trade names, model number, instrument identification number, diameters, material composition, etc.

DATA REPORT

Data reports are to include laboratory analytical results, QA/QC information, field indicator parameters measured during purging, field instrument calibration information, and whatever other field logbook information is needed to allow for a full evaluation of data usability.

Note: the use of trade, product, or firm names in this sampling procedure is for descriptive purposes only and does not constitute endorsement by the U.S. EPA.

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APPENDIX A
PERISTALTIC PUMPS

Before selecting a peristaltic pump to collect groundwater samples for VOCs and/or dissolved gases (e.g., methane, carbon dioxide, etc.) consideration should be given to the following:

- The decision of whether or not to use a peristaltic pump is dependent on the intended use of the data.
- If the additional sampling error that may be introduced by this device is NOT of concern for the VOC/dissolved gases data's intended use, then this device may be acceptable.
- If minor differences in the groundwater concentrations could effect the decision, such as to continue or terminate groundwater cleanup or whether the cleanup goals have been reached, then this device should NOT be used for VOC/dissolved gases sampling. In these cases, centrifugal or bladder pumps are a better choice for more accurate results.

EPA and USGS have documented their concerns with the use of the peristaltic pumps to collect water sample in the below documents.

- "Suction Pumps are not recommended because they may cause degassing, pH modification, and loss of volatile compounds" *A Compendium of Superfund Field Operations Methods*, EPA/540/P-87/001, December 1987.
- "The agency does not recommend the use of peristaltic pumps to sample ground water particularly for volatile organic analytes" *RCRA Ground-Water Monitoring Draft Technical Guidance*, EPA Office of Solid Waste, November 1992.
- "The peristaltic pump is limited to shallow applications and can cause degassing resulting in alteration of pH, alkalinity, and volatiles loss", *Low-flow (Minimal drawdown) Ground-Water Sampling Procedures*, by Robert Puls & Michael Barcelona, April 1996, EPA/540/S-95/504.
- "Suction-lift pumps, such as peristaltic pumps, can operate at a very low pumping rate; however, using negative pressure to lift the sample can result in the loss of volatile analytes", USGS Book 9 Techniques of Water-Resources Investigation, Chapter A4. (Version 2.0, 9/2006).

APPENDIX B

SUMMARY OF SAMPLING INSTRUCTIONS

These instructions are for using an adjustable rate, submersible pump or a peristaltic pump with the pump's intake placed at the midpoint of a 10 foot or less well screen or an open interval. The water level in the monitoring well is above the top of the well screen or open interval, the ambient temperature is above 32°F, and the equipment is not dedicated. Field instruments are already calibrated. The equipment is setup according to the diagram at the end of these instructions.

1. Review well installation information. Record well depth, length of screen or open interval, and depth to top of the well screen. Determine the pump's intake depth (e.g., mid-point of screen/open interval).

2. On the day of sampling, check security of the well casing, perform any safety checks needed for the site, lay out a sheet of polyethylene around the well (if necessary), and setup the equipment. If necessary a canopy or an equivalent item can be setup to shade the pump's tubing and flow-through-cell from the sun light to prevent the sun light from heating the groundwater.

3. Check well casing for a reference mark. If missing, make a reference mark. Measure the water level (initial) to 0.01 ft. and record this information.

4. Install the pump's intake to the appropriate depth (e.g., midpoint) of the well screen or open interval. Do not turn-on the pump at this time.

5. Measure water level and record this information.

6. Turn-on the pump and discharge the groundwater into a graduated waste bucket. Slowly increase the flow rate until the water level starts to drop. Reduce the flow rate slightly so the water level stabilizes. Record the pump's settings. Calculate the flow rate using a graduated container and a stop watch. Record the flow rate. Do not let the water level drop below the top of the well screen.

If the groundwater is highly turbid or colored, continue to discharge the water into the bucket until the water clears (visual observation); this usually takes a few minutes. The turbid or colored water is usually from the well being disturbed during the pump installation. If the water does not clear, then you need to make a choice whether to continue purging the well (hoping that it will clear after a reasonable time) or continue to

the next step. Note, it is sometimes helpful to install the pump the day before the sampling event so that the disturbed materials in the well can settle out.

If the water level drops to the top of the well screen during the purging of the well, stop purging the well, and do the following:

> Wait for the well to recharge to a sufficient volume so samples can be collected. This may take awhile (pump maybe removed from well, if turbidity is not a problem). The project manager will need to make the decision when samples should be collected and the reasons recorded in the site's log book. A water level measurement needs to be performed and recorded before samples are collected. When samples are being collected, the water level must not drop below the top of the screen or open interval. Collect the samples from the pump's tubing. Always collect the VOCs and dissolved gases samples first. Normally, the samples requiring a small volume are collected before the large volume samples are collected just in case there is not sufficient water in the well to fill all the sample containers. All samples must be collected, preserved, and stored according to the analytical method. Remove the pump from the well and decontaminate the sampling equipment.

If the water level has dropped 0.3 feet or less from the initial water level (water level measure before the pump was installed); proceed to Step 7. If the water level has dropped more than 0.3 feet, calculate the volume of water between the initial water level and the stabilized water level. Add the volume of the water which occupies the pump's tubing to this calculation. This combined volume of water needs to be purged from the well after the water level has stabilized before samples are be collected.

7. Attach the pump's tubing to the "T" connector with a valve (or a three-way stop cock). The pump's tubing from the well casing to the "T" connector must be as short as possible to prevent the groundwater in the tubing from heating up from the sun light or from the ambient air. Attach a short piece of tubing to the other end of the end of the "T" connector to serve as a sampling port for the turbidity samples. Attach the remaining end of the "T" connector to a short piece of tubing and connect the tubing to the flow-through-cell bottom port. To the top port, attach a small piece of tubing to direct the water into a calibrated waste bucket. Fill the cell with the groundwater and remove all gas bubbles from the cell. Position the flow-through-cell in such a way that if gas bubbles enter the cell they can easily exit the cell. If the ports are on the same side of the cell and the cell is cylindrical shape, the cell can be placed at a 45-degree angle with the ports facing upwards; this position should keep any gas bubbles entering the cell away from the monitoring probes and allow the gas bubbles to exit the cell easily (see Low-Flow Setup Diagram). Note,

make sure there are no gas bubbles caught in the probes' protective guard; you may need to shake the cell to remove these bubbles.

8. Turn-on the monitoring probes and turbidity meter.

9. Record the temperature, pH, dissolved oxygen, specific conductance, and oxidation/reduction potential measurements. Open the valve on the "T" connector to collect a sample for the turbidity measurement, close the valve, do the measurement, and record this measurement. Calculate the pump's flow rate from the water exiting the flow-through-cell using a graduated container and a stop watch, and record the measurement. Measure and record the water level. Check flow-through-cell for gas bubbles and sediment; if present, remove them.

10. Repeat Step 9 every 5 minutes or as appropriate until monitoring parameters stabilized. Note at least one flow-through-cell volume must be exchanged between readings. If not, the time interval between readings will need to be increased. Stabilization is achieved when three consecutive measurements are within the following limits:

Turbidity (10% for values greater than 5 NTUs; if three Turbidity values are less than 5 NTUs, consider the values as stabilized),
Dissolved Oxygen (10% for values greater than 0.5 mg/L, if three Dissolved Oxygen values are less than 0.5 mg/L, consider the values as stabilized),
Specific Conductance (3%),
Temperature (3%),
pH (± 0.1 unit),
Oxidation/Reduction Potential (±10 millivolts).

If these stabilization requirements do not stabilize in a reasonable time, the probes may have been coated from the materials in the groundwater, from a buildup of sediment in the flow-through-cell, or a gas bubble is lodged in the probe. The cell and the probes will need to be cleaned. Turn-off the probes (not the pump), disconnect the cell from the "T" connector and continue to purge the well. Disassemble the cell, remove the sediment, and clean the probes according to the manufacturer's instructions. Reassemble the cell and connect the cell to the "T" connector. Remove all gas bubbles from the cell, turn-on the probes, and continue the measurements. Record that the time the cell was cleaned.

11. When it is time to collect the groundwater samples, turn-off the monitoring probes, and disconnect the pump's tubing from the "T" connector. If you are using a centrifugal or peristaltic pump check the pump's tubing to determine if the tubing is completely filled with water (no air space).

All samples must be collected and preserved according to the analytical method. VOCs and dissolved gases samples are normally collected first and directly into pre-preserved sample containers. However, this may not be the case for all sampling locations; the SAP/QAPP should list the order in which the samples are to be collected based on the project's objective(s). Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

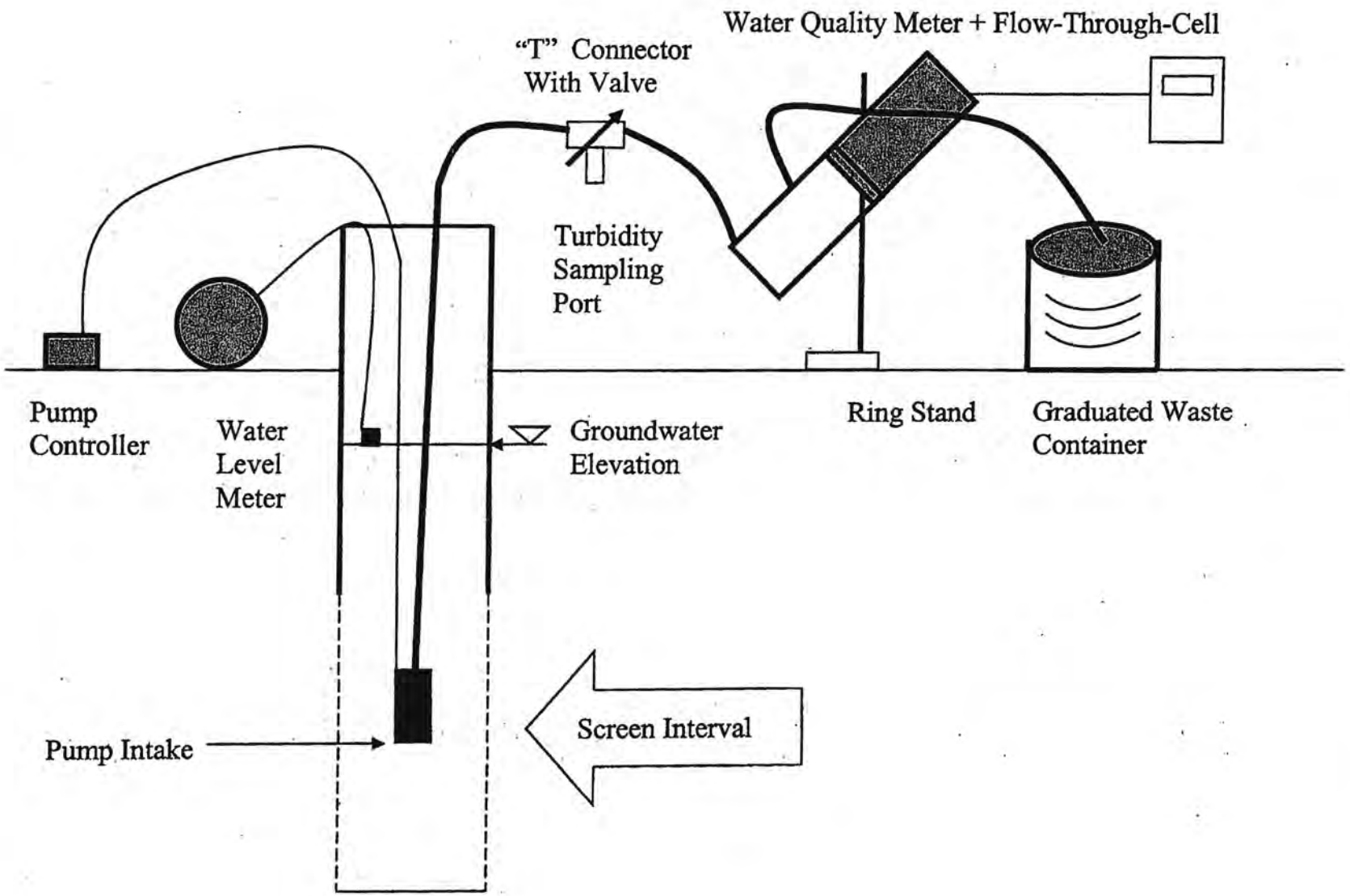
If the pump's tubing is not completely filled with water and the samples are being collected for VOCs and/or dissolved gases analyses using a centrifugal or peristaltic pump, do the following:

> All samples must be collected and preserved according to the analytical method. The VOCs and the dissolved gases (e.g., methane, ethane, ethene, and carbon dioxide) samples are collected last. When it becomes time to collect these samples increase the pump's flow rate until the tubing is completely filled. Collect the samples and record the new flow rate.

12. Store the samples according to the analytical method.

13. Record the total purged volume (graduated waste bucket). Remove the pump from the well and decontaminate the sampling equipment.

Low-Flow Setup Diagram



APPENDIX C
EXAMPLE (Minimum Requirements)
WELL PURGING-FIELD WATER QUALITY MEASUREMENTS FORM

Location (Site/Facility Name)_____
Well Number_____Date_____
Field Personnel_____
Sampling Organization_____
Identify MP_____

Depth to _____/_____ of screen
(below MP) top bottom
Pump Intake at (ft. below MP)_____
Purging Device; (pump type)_____
Total Volume Purged _____

Clock Time 24 HR	Water Depth below MP ft	Pump Dial[1]	Purge Rate ml/min	Cum. Volume Purged liters	Temp. °C	Spec. Cond.[2] µS/cm	pH	ORP[3] mv	DO mg/L	Turb-idity NTU	Comments
Stabilization Criteria					3%	3%	±0.1	± 10 mv	10%	10%	

1. Pump dial setting (for example: hertz, cycles/min, etc).
2. µSiemens per cm(same as µmhos/cm)at 25°C.
3. Oxidation reduction potential (ORP)

STANDARD OPERATING PROCEDURE
CALIBRATION OF FIELD INSTRUMENTS
(temperature, pH, dissolved oxygen, conductivity/specific conductance, oxidation/reduction potential [ORP], and turbidity)

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Prepared by: Charles Porfert 1/19/10
(Charles Porfert, Quality Assurance Unit) Date

Approved by: Gerard Sotolongo 1-19-10
(Gerard Sotolongo, Quality Assurance Unit) Date

Revision Page

Date	Rev #	Summary of changes	Sections
6/03/98	1	Draft	
01/19/10	2	Finalized	

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1.0 SCOPE AND APPLICATION

The purpose of this standard operating procedure (SOP) is to provide a framework for calibrating field instruments used to measure water quality parameters for groundwater and surface water. Water quality parameters include temperature, pH, dissolved oxygen, specific conductance, oxidation/reduction potential [ORP], and turbidity. This SOP supplements, but does not replace, EPA analytical methods listed in 40 CFR 136 and 40 CFR 141 for temperature, dissolved oxygen, conductivity/specific conductance, pH and turbidity.

This SOP is written for instruments that measure temperature, pH, dissolved oxygen, specific conductance, turbidity, and/or oxidation/reduction potential [ORP] and the probe readings for pH, dissolved oxygen, and specific conductance are automatically corrected for temperature.

For groundwater monitoring, the instrument must be equipped with a flow-through-cell and the display/logger or computer display screen needs to be large enough to simultaneously contain the readouts of each probe in the instrument. Turbidity is measured using a separate instrument. It must not be measured in a flow-through-cell because the flow-through-cell acts as a sediment trap. This procedure is applicable for use with the *EPA Region 1 Low Stress (low flow) Purging and Sampling Procedure for the Collection of Ground Water Samples from Monitoring Wells.*

2.0 HEALTH AND SAFETY WARNINGS

Read all labels on the standards and note any warnings on the labels. Wear appropriate personal protection equipment (e.g., gloves, eye shields, etc.) when handling the standards. If necessary, consult the Material Safety Data Sheets (MSDS) for additional safety information on the chemicals in the standards.

3.0 GENERAL

All monitoring instruments must be calibrated before they are used to measure environmental samples. For instrument probes that rely on the temperature sensor (pH, dissolved oxygen, specific conductance, and oxidation/reduction potential [ORP]), each temperature sensor needs to be checked for accuracy against a thermometer that is traceable to the National Institute of Standards and Technology (NIST). Before any instrument is calibrated or used to perform environmental measurements, the instrument must stabilize (warm-up) according to manufacturer's instructions and must have no air bubbles lodged between the probe and probe guard.

Most projects will require at least two standards to bracket the expected measurement range. This means that one standard is less than the expected value and one is higher. When an environmental sample measurement falls outside the calibration range, the instrument must be re-calibrated to bracket the new range before continuing measurements. Otherwise, the measurements that are outside the calibration range will need to be qualified.

This SOP requires that the manufacturer's instruction manual (including the instrument specifications) accompany the instrument into the field.

4.0 FREQUENCY OF CALIBRATION

At a minimum, the instrument is calibrated prior to use on the day the measurements are to be performed. A post calibration check at the end of the day is performed to determine if the instrument drifted out of calibration. Some projects may require more frequent calibration checks throughout the day in addition to the check at the end of the day. For these checks, the instrument can be recalibrated during the day if the instrument drifted out of calibration and only the data measured prior to the check would need to be qualified. The calibration/post calibration data information is recorded in Table 1.

Instruments (e.g., sonde) that monitor continuously over a period of time are calibrated before deployment. When these instruments are recovered, the calibration is checked to determine if any of them drifted out of calibration.

Some instruments lose their calibration criteria when they are turned off. Those instruments can either be left on all day (battery dependent) or calibrated at each sampling location. If they are calibrated at each sampling location, a post calibration check is not needed.

Ideally, the temperature of the standards should be close to the temperature of the ambient water that is being measured.

5.0 CALIBRATION PROCEDURES

Prior to calibration, all instrument probes and cable connections must be cleaned and the battery checked according to the manufacturer's instructions. Failure to perform these steps (proper maintenance) can lead to erratic measurements.

If a multi-probe instrument is to be used, program the instrument to display the parameters to be measured (e.g., temperature, pH, percent dissolved oxygen, mg/L dissolved oxygen, specific conductance, and ORP).

The volume of the calibration solutions must be sufficient to cover both the probe and temperature sensor (see manufacturer's instructions for the volume to be used).

Check the expiration date of the standards. Do not use expired standards.

All standards are stored according to manufacturer instructions.

5.1 TEMPERATURE

Most instrument manuals state there is no calibration of the temperature sensor, but the temperature sensor must be checked to determine its accuracy. This accuracy check is performed at least once per year and the accuracy check date/information is kept with the instrument. If the accuracy check date/information is not included with the instrument or the last check was over a year, the temperature sensor accuracy needs to be checked at the beginning of the sampling event. If the instrument contains multiple temperature sensors, each sensor must be checked. This procedure is not normally perform in the field. If the instrument is obtained from a rental company, the rental company should performed the calibration check and include with the instrument documentation that it was performed.

Verification Procedure

1. Fill a container with water and adjust the water temperature to below the water body's temperature to be measured. Use ice or warm water to adjust the temperature.

2. Place a thermometer that is traceable to the National Institute of Standards and Technology (NIST) and the instrument's temperature sensor into the water. Wait for both temperature readings to stabilize.

3. Compare the two measurements. The instrument's temperature sensor must agree with the reference thermometer measurement within the accuracy of the sensor (e.g., ±0.2°C). If the measurements do not agree, the instrument may not be working properly and the manufacturer needs to be consulted.

4. Adjust the water temperature to a temperature higher than the water body to be measured.

5. Compare the two measurements. The instrument's temperature sensor must agree with the reference thermometer measurement within the accuracy of the sensor (e.g.,

±0.2°C). If the measurements do not agree, the instrument may not be working properly and the manufacturer needs to be consulted.

5.2 pH (electrometric)

The pH of a sample is determined electrometrically using a glass electrode.

Choose the appropriate buffered standards that will bracket the expected values at the sampling locations. If the water body's pH is unknown, then three standards are needed for the calibration: one close to seven, one at least two pH units below seven, and the other at least two pH units above seven. Instruments that will not accept three standards will need to be re-calibrated if the water sample's pH is outside the initial calibration range described by the two standards.

Calibration Procedure

1. Allow the buffered standards to equilibrate to the ambient temperature.
2. Fill calibration containers with the buffered standards so each standard will cover the pH probe and temperature sensor.
3. Remove probe from its storage container, rinse with deionized water, and remove excess water.
4. Select measurement mode. Immerse probe into the initial standard (e.g., pH 7).
5. Wait until the readings stabilize. If the reading does not change within 30 seconds, select calibration mode and then select "pH". Enter the buffered standard value into instrument.
6. Remove probe from the initial standard, rinse with deionized water, and remove excess water.
7. Immerse probe into the second standard (e.g., pH 4). Repeat step 5.
8. Remove probe from the second standard, rinse with deionized water, and remove excess water. If instrument only accepts two standards, the calibration is complete. Go to step 11. Otherwise continue.
9. Immerse probe in third buffered standard (e.g., pH 10) and repeat step 5.

10. Remove probe from the third standard, rinse with deionized water, and remove excess water.

11. Select measurement mode, if not already selected. To ensure that the initial calibration standard (e.g., pH 7) has not changed, immerse the probe into the initial standard. Wait for the readings to stabilize. The reading should read the initial standard value within the manufacturer's specifications. If not, re-calibrate the instrument. If re-calibration does not help, the calibration range may be too great. Reduce calibration range by using standards that are closer together.

12. The calibration is complete. Rinse the probe with deionized water and store the probe according to manufacturer's instructions.

13. Record the calibration information on Table 1.

5.3 DISSOLVED OXYGEN

Dissolved oxygen (DO) content in water is measured using a membrane electrode. To insure proper operation, the DO probe's membrane and electrolyte should be replaced prior to calibration for the sampling event. The new membrane may need to be conditioned before it is used; consult manufacturer's manual on how the conditioning is to be performed. Failure to perform this step may lead to erratic measurements. Before performing the calibration/measurements, inspect the membrane for air bubbles and nicks.

Note: some manufacturers require an altitude correction instead of a barometric correction. In that case, enter the altitude correction according to the manufacturer's directions in Step 5 and then proceed to Step 6.

Note: some instruments have a built-in barometer. Follow the manufacturer's instructions for entering the barometric value in step 5.

Calibration Procedure

1. Gently dry the temperature sensor and remove any water droplets from the DO probe's sensor membrane according to manufacturer's instructions. Note that the evaporation of moisture on the temperature sensor or DO probe may influence the readings during calibration.

2. Create a 100 percent water-saturated air environment by placing a wet sponge or a

wet paper towel on the bottom of the DO calibration container. Place the DO probe into the calibration container. The probe is loosely fitted into the calibration container to prevent the escape of moisture evaporating from the sponge or paper towel while maintaining ambient pressure (see manufacturer's instructions). Note that the probe and the temperature sensor must not come in contact with these wet items.

3. Allow the confined air to become saturated with water vapor (saturation occurs in approximately 10 to 15 minutes). During this time, turn on the instrument to allow the DO probe to warm-up. Select the measurement mode. Check the temperature readings. Readings must stabilize before continuing to the next step.

4. Select calibration mode; then select "DO %".

5. Enter the local barometric pressure (usually in mm of mercury) for the sampling location into the instrument. This measurement must be determined from an on-site barometer. Do not use barometric pressure obtained from the local weather services unless the pressure is corrected for the elevation of the sampling location. [Note: inches of mercury times 25.4 mm/inch equals mm of mercury or consult Oxygen Solubility at Indicated Pressure chart attached to the SOP for conversion at selected pressures].

6. The instrument should indicate that the calibration is in progress. After calibration, the instrument should display percent saturated DO.

7. Select measurement mode and set the display to read DO mg/L and temperature. Compare the DO mg/L reading to the Oxygen Solubility at Indicated Pressure chart attached to the SOP. The numbers should agree. If they do not agree within the accuracy of the instrument (usually ± 0.2 mg/L), repeat calibration. If this does not work, change the membrane and electrolyte solution.

8. Remove the probe from the container and place it into a 0.0 mg/L DO solution (see footnote). Check temperature readings. They must stabilize before continuing.

9. Wait until the "mg/L DO" readings have stabilized. The instrument should read less than 0.5 mg/L (assuming an accuracy of ± 0.2 mg/L). If the instrument reads above 0.5 mg/L or reads negative, it will be necessary to clean the probe, and change the membrane and electrolyte solution. If this does not work, try a new 0.0 mg/L DO solution. If these changes do not work, contact the manufacturer. Note: some projects and instruments may have different accuracy requirements. The 0.5 mg/L

value may need to be adjusted based on the accuracy requirements of the project or instrument.

10. After the calibration has been completed, rinse the probe with tap or deionized water and store the probe according to manufacturer's instructions. It is important that all of the 0.0 mg/L DO solution be rinsed off the probe so as not to effect the measurement of environmental samples.

11. Record calibration information on Table 1.

Note: You can either purchase the 0.0 mg/L DO solution from a vendor or prepare the solution yourself. To prepare a 0.0 mg/L DO solution, follow the procedure stated in Standard Methods (Method 4500-O G). The method basically states to add excess sodium sulfite (until no more dissolves) and a trace amount of cobalt chloride (read warning on the label before use) to water. This solution is prepared prior to the sampling event. Note: this solution can be made without cobalt chloride, but the probe will take longer to respond to the low DO concentration.

5.4 SPECIFIC CONDUCTANCE

Conductivity is used to measure the ability of an aqueous solution to carry an electrical current. Specific conductance is the conductivity value corrected to 25 °C.

Most instruments are calibrated against a single standard which is near the specific conductance of the environmental samples. The standard can be either below or above the specific conductance of the environmental samples. A second standard is used to check the linearity of the instrument in the range of measurements.

When performing specific conductance measurement on groundwater or surface water and the measurement is outside the initial calibration range defined by the two standards, the instrument will need to be re-calibrated using the appropriate standards.

Specific Conductance Calibration Procedure

1. Allow the calibration standards to equilibrate to the ambient temperature.

2. Fill calibration containers with the standards so each standard will cover the probe and temperature sensor. Remove probe from its storage container, rinse the probe with deionized water or a small amount of the standard (discard the rinsate), and place the

probe into the standard.

3. Select measurement mode. Wait until the probe temperature has stabilized.

4. Select calibration mode, then specific conductance. Enter the specific conductance standard value. Make sure that the units on the standard are the same as the units used by the instrument. If not, convert the units on the standard to the units used by the instrument.

5. Select measurement mode. The reading should remain within manufacturer's specifications. If it does not, re-calibrate. If readings continue to change after re-calibration, consult manufacturer or replace calibration solution.

6. Remove probe from the standard, rinse the probe with deionized water or a small amount of the second standard (discard the rinsate), and place the probe into the second standard. The second standard will serve to verify the linearity of the instrument. Read the specific conductance value from the instrument and compare the value to the specific conductance on the standard. The two values should agree within the specifications of the instrument. If they do not agree, re-calibrate. If readings do not compare, then the second standard may be outside the linear range of the instrument. Use a standard that is closer to the first standard and repeat the verification. If values still do not compare, try cleaning the probe or consult the manufacturer.

7. After the calibration has been completed, rinse the probe with deionized water and store the probe according to manufacturer's instructions.

8. Record the calibration information on Table 1.

Note: for projects where specific conductance is not a critical measurement it may be possible to calibrate with one standard in the range of the expected measurement.

5.5 OXIDATION/REDUCTION POTENTIAL (ORP)

The oxidation/reduction potential is the electrometric difference measured in a solution between an inert indicator electrode and a suitable reference electrode. The electrometric difference is measured in millivolts and is temperature dependent.

Calibration or Verification Procedure

1. Allow the calibration standard (a Zobell solution: read the warning on the label before use) to equilibrate to ambient temperature.

2. Remove the probe from its storage container and place it into the standard.

3. Select measurement mode.

4. Wait for the probe temperature to stabilize, and then read the temperature.

5. If the instrument is to be calibrated, do Steps 6 and 7. If the instrument calibration is to be verified, then go to Step 8.

6. Look up the millivolt (mv) value at this temperature from the millivolt versus temperature correction table usually found on the standard bottle or on the standard instruction sheet. You may need to interpolate millivolt value between temperatures. Select "calibration mode", then "ORP". Enter the temperature-corrected ORP value into the instrument.

7. Select measurement mode. The readings should remain unchanged within manufacturer's specifications. If they change, re-calibrate. If readings continue to change after re-calibration, try a new Zobell solution or consult manufacturer. Go to Step 9.

8. If the instrument instruction manual states that the instrument is factory calibrated, then verify the factory calibration against the Zobell solution. If they do not agree within the specifications of the instrument, try a new Zobell solution. If it does not agree, the instrument will need to be re-calibrated by the manufacturer.

9. After the calibration has been completed, rinse the probe with deionized water and store the probe according to manufacturer's instructions.

10. Record the calibration information on Table 1.

5.6 TURBIDITY

The turbidity method is based upon a comparison of intensity of light scattered by a sample under defined conditions with the intensity of light scattered by a standard reference suspension. A

turbidimeter is a nephelometer with a visible light source for illuminating the sample and one or more photo-electric detectors placed ninety degrees to the path of the light source. Note: the below calibration procedure is for a turbidimeter which the sample is placed into a cuvette.

Some instruments will only accept one standard. For those instruments, the second, third, etc., standards will serve as check points.

Calibration Procedures

1. Allow the calibration standards to equilibrate at the ambient temperature. The use of commercially available polymer primary standards (AMCO-AEPA-1) is preferred; however, the standards can be prepared using Formazin (read the warning on the label before use) according to the EPA analytical Method 180.1. Other standards may be used if they can be shown that they are equivalent to the previously mentioned standards.

2. If the standard cuvette is not sealed, rinse a cuvette with deionized water. Shake the cuvette to remove as much water as possible. Do not wipe dry the inside of the cuvette because lint from the wipe may remain in the cuvette. Add the standard to the cuvette.

3. Before performing the calibration procedure, make sure the cuvettes are not scratched and the outside surfaces are dry and free from fingerprints and dust. If the cuvette is scratched or dirty, discard or clean the cuvette respectively. Note: some manufacturers require the cuvette to be orientated in the instrument in a particular direction for accurate reading.

4. Select a low value standard such as a zero or 0.02 NTU and calibrate according to manufacturer's instructions. Note: a zero standard (approximately 0 NTU) can be prepared by passing distilled water through a 0.45 micron pore size membrane filter.

5. Select a high standard and calibrate according to manufacturer's instructions or verify the calibration if instrument will not accept a second standard. In verifying, the instrument should read the standard value to within the specifications of the instrument. If the instrument has range of scales, check each range that will be used during the sampling event with a standard that falls within that range.

6. Record the calibration information on Table 1.

6.0 POST CALIBRATION CHECK

After the initial calibration is performed, the instrument's calibration may drift during the measurement period. As a result, you need to determine the amount of drift that occurred after collecting the measurements. This is performed by placing the instrument in measurement mode (not calibration mode) and placing the probe in one or more of the standards used during the initial calibration; for turbidity place the standard in a cuvette and then into the turbidimeter. Wait for the instrument to stabilize and record the measurement (Table 1). Compare the measurement value to the initial calibration value. This difference in value is then compared to the drift criteria or post calibration criteria described in the quality assurance project plan or the sampling and analysis plan for the project. If the check value is outside the criteria, then the measurement data will need to be qualified.

For the dissolved oxygen calibration check, follow the calibration instructions steps one through three while the instrument is in measurement mode. Record dissolved oxygen value (mg/L), temperature, and barometric pressure. Compare the measurement value to the Oxygen Solubility at Indicated Pressure chart attached to this SOP. The value should be within the criteria specified for the project. If measurement value drifted outside the criteria, the data will need to be qualified.

If the quality assurance project plan or the sampling and analysis plan do not list the drift criteria or the post-calibration criteria, use the criteria below.

Measurement	**Post Calibration Criteria**
Dissolved Oxygen	± 0.5 mg/L of sat. value* < 0.5 mg/L for the 0 mg/L solution, but not a negative value
Specific Conductance	±5% of standard or ± 10 µS/cm (whichever is greater)
pH	± 0.3 pH unit with pH 7 buffer*
Turbidity	± 5% of standard
ORP	± 10 mv*

Note: * Table 8.1, USEPA Region 1 *YSI 6-Series Sondes and Data Logger SOP*, January 30, 2007, revision 9.

7.0 DATA MANAGEMENT AND RECORDS MANAGEMENT

All calibration records must be documented in the project's log book or on a calibration log sheet. At a minimum, include the instrument manufacturer, model number, instrument identification number (when more than one instrument of the same model is used), the standards used to calibrate the instruments (including source), the calibration date, the instrument readings, the post calibration check, and the name of the person(s) who performed the calibration. An example of a calibration log sheet is shown in Table 1.

8.0 References

Standard Methods for the Examination of Water and Wastewater, 20th edition, 1998.

Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March 1983.

Turbidity - Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, August 1993.

USEPA Region 1 YSI 6-Series Sondes and Data Logger SOP, January 30, 2007, revision 9.

USGS Guidelines and Standard Procedures for Continuous Water-Quality Monitors: Station Operation, Record Computation, and Data Reporting, Techniques and Methods 1-D3.

TABLE 1
INSTRUMENT CALIBRATION LOG

Project Name_____ Date_____
Weather_____

Calibrated by_____ Instrument_____
Serial Number_____

Parameters	Morning Calibration	Morning Temperature	End of Day Calibration Check*	End of Day Temperature
Specific Conductance Standard #1				
Specific Conductance Standard #2				
pH (7)				
pH (4)				
pH (10)				
ORP Zobel solution				
Dissolved Oxygen 100% water saturated air mg/L				
Dissolved Oxygen Zero Dissolved Oxygen Solution mg/L				
Barometric Pressure mm Hg		NA		NA
Turbidity Standard #1				
Turbidity Standard #2				
Turbidity Standard #3				

* For each Parameter, chose one standard as your check standard. If possible, choose the one that is closest to the ambient measurement value.

Oxygen Solubility at Indicated Pressure

Temp.	Pressure (Hg)							
	760	755	750	745	740	735	730	mm
°C	29.92	29.72	29.53	29.33	29.13	28.94	28.74	in
0	14.57	14.47	14.38	14.28	14.18	14.09	13.99	mg/l
1	14.17	14.08	13.98	13.89	13.79	13.70	13.61	
2	13.79	13.70	13.61	13.52	13.42	13.33	13.24	
3	13.43	13.34	13.25	13.16	13.07	12-98	12.90	
4	13.08	12.99	12.91	12.82	12.73	12.65	12.56	
5	12.74	12.66	12.57	12.49	12.40	12.32	12.23	
6	12.42	12.34	12.26	12.17	12.09	12.01	11.93	
7	12.11	12.03	11.95	11.87	11.79	11.71	11.63	
8	11.81	11.73	11.65	11.57	11.50	11.42	11.34	
9	11.53	11.45	11.38	11.30	11.22	11.15	11.07	
10	11.28	11.19	11.11	11.04	10.96	10.89	10.81	
11	10.99	10.92	10.84	10.77	10.70	10.62	10.55	
12	10.74	10.67	10.60	10.53	10.45	10.38	10.31	
13	10.50	10.43	10.36	10.29	10.22	10.15	10.08	
14	10.27	10.20	10.13	10.06	10.00	9.93	9.86	
15	10.05	9.98	9.92	9.85	9.78	9.71	9.65	
16	9.83	9.76	9.70	9.63	9.57	9.50	9.43	
17	9.63	9.57	9.50	9.44	9.37	9.31	9.24	
18	9.43	9.37	9.30	9.24	9.18	9.11	9.05	
19	9.24	9.18	9.12	9.05	8.99	8.93	8.87	
20	9.06	9.00	8.94	8.88	8.82	8.75	8.69	
21	8.88	8.82	8.76	8.70	8.64	8.58	8.52	
22	8.71	8.65	8.59	8.53	8.47	8.42	8.36	
23	8.55	8.49	8.43	8.38	8.32	8.26	8.20	
24	8.39	8.33	8.28	8.22	8.16	8.11	8.05	
25	8.24	8.18	8.13	8.07	8.02	7.96	7.90	
26	8.09	8.03	7.98	7.92	7.87	7.81	7.76	
27	7.95	7.90	7.84	7.79	7.73	7.68	7.62	
28	7.81	7.76	7.70	7.65	7.60	7.54	7.49	
29	7.68	7.63	7.57	7.52	7.47	7.42	7.36	
30	7.55	7.50	7.45	7.39	7.34	7.29	7.24	
31	7.42	7.37	7.32	7.27	7.22	7.16	7.11	
32	7.30	7.25	7.20	7.15	7.10	7.05	7.00	
33	7.08	7.13	7.08	7.03	6.98	6.93	6.88	
34	7.07	7.02	6.97	6.92	6.87	6.82	6.78	
35	6.95	6.90	6.85	6.80	6.76	6.71	6.66	
36	6.84	6.79	6.76	6.70	6.65	6.60	6.55	
37	6.73	6.68	6.64	6.59	6.54	6.49	6.45	
38	6.63	6.58	6.54	6.49	6.44	6.40	6.35	
39	6.52	6.47	6.43	6.38	6.35	6.29	6.24	
40	6.42	6.37	6.33	6.28	6.24	6.19	6.15	
41	6.32	6.27	6.23	6.18	6.14	6.09	6.05	
42	6.22	6.18	6.13	6.09	6.04	6.00	5.95	
43	6.13	6.09	6.04	6.00	5.95	5.91	5.87	
44	6.03	5.99	5.94	5.90	5.86	5.81	5.77	
45	5.94	5.90	5.85	5.81	5.77	5.72	5.68	

(Continued)

Source: Draft EPA Handbook of Methods for Acid Deposition Studies, Field Operations for Surface Water Chemistry, EPA/600/4-89/020, August 1989.

Oxygen Solubility at Indicated Pressure (continued)

Temp.	Pressure (Hg)								
	725	720	715	710	705	700	695	690	mm
°C	28.54	28.35	28.15	27.95	27.76	27.56	27.36	27.17	in
0	13.89	13.80	13.70	13.61	13.51	13.41	13.32	13.22	mg/l
1	13.51	13.42	13.33	13.23	13.14	13.04	12.95	12.86	
2	13.15	13.06	12.07	12.88	12.79	12.69	12.60	12.51	
3	12.81	12.72	12.63	12.54	12.45	12.36	12.27	12.18	
4	12.47	12.39	12.30	12.21	12.13	12.04	11.95	11.87	
5	12.15	12.06	11.98	11.89	11.81	11.73	11.64	11.56	
6	11.84	11.73	11.68	11.60	11.51	11.43	11.35	11.27	
7	11.55	11.47	11.39	11.31	11.22	11.14	11.06	10.98	
8	11.26	11.18	11.10	11.02	10.95	10.87	10.79	10.71	
9	10.99	10.92	10.84	10.76	10.69	10.61	10.53	10.46	
10	10.74	10.66	10.59	10.51	10.44	10.36	10.29	10.21	
11	10.48	10.40	10.33	10.28	10.18	10.11	10.04	9.96	
12	10.24	10.17	10.10	10.02	9.95	9.88	9.81	9.46	
13	10.01	9.94	9.87	9.80	9.73	9.66	9.59	9.52	
14	9.79	9.72	9.65	9.68	9.51	9.45	9.38	9.31	
15	9.58	9.51	9.44	9.58	9.31	9.24	9.18	9.11	
16	9.37	9.30	9.24	9.17	9.11	9.04	8.97	8.91	
17	9.18	9.11	9.05	8.98	8.92	8.85	8.79	8.73	
18	8.99	8.92	8.86	8.80	8.73	8.67	8.61	8.54	
19	8.81	8.74	8.68	8.62	8.56	8.49	8.43	8.37	
20	8.63	8.57	8.51	8.45	8.39	8.33	8.27	8.21	
21	8.46	8.40	8.34	8.28	8.22	8.16	8.10	8.04	
22	8.30	8.24	8.18	8.12	8.06	8.00	7.95	7.89	
23	8.15	8.09	8.03	7.97	7.91	7.86	7.80	7.74	
24	7.99	7.94	7.88	7.82	7.76	7.71	7.65	7.59	
25	7.85	7.79	7.74	7.68	7.60	7.57	7.51	7.46	
26	7.70	7.65	7.59	7.54	7.48	7.43	7.37	7.32	
27	7.57	7.52	7.46	7.41	7.35	7.30	7.25	7.19	
28	7.44	7.38	7.33	7.28	7.22	7.17	7.12	7.06	
29	7.31	7.26	7.21	7.15	7.10	7.05	7.00	6.94	
30	7.19	7.14	7.08	7.03	6.98	6.93	6.88	6.82	
31	7.06	7.01	6.96	6.91	6.86	6.81	6.76	6.70	
32	6.95	6.90	6.85	6.80	6.70	6.70	6.64	6.59	
33	6.83	6.78	6.73	6.68	6.83	6.58	6.53	6.48	
34	6.73	6.68	6.63	6.58	6.53	6.48	6.43	6.38	
35	6.61	6.56	6.51	6.47	6.42	6.37	6.36	6.27	
36	6.51	6.46	6.41	6.36	6.31	6.27	6.22	6.17	
37	6.40	6.35	6.31	6.26	6.21	6.16	6.12	6.07	
38	6.30	6.26	6.21	6.16	6.12	6.07	6.02	5.98	
39	6.26	6.15	6.11	6.06	6.01	5.97	5.92	5.87	
40	6.10	6.06	6.01	5.96	5.92	5.86	5.83	5.78	
41	6.00	5.96	5.91	5.87	5.82	5.78	5.73	5.69	
42	5.91	5.86	5.82	5.77	5.73	5.69	5.64	5.60	
43	5.82	5.78	5.73	5.69	5.65	5.60	5.56	5.51	
44	5.72	5.68	5.64	5.59	5.55	5.51	5.46	5.42	
45	5.64	5.59	5.55	5.51	5.47	5.42	5.38	5.34	

Source: Draft EPA Handbook of Methods for Acid Deposition Studies, Field Operations for Surface Water Chemistry, EPA/600/4-89/020, August 1989.

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		Prepared Earth Sciences Department	
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1.0 PURPOSE

The purpose of this guideline is to provide a general description of, and technical management guidance on, the use of Magnetic and Metal Detection Surveys for site investigations.

2.0 SCOPE

This guideline provides a description of the principles of operation, instrumentation, applicability, and implementability of standard geophysical methods used during site investigations to determine site features related to magnetic anomalies and buried metal. This document is intended to be used by the project manager, field operations leader, or site geologist to develop a sufficient understanding of each method and to assist in proper work plan development and scheduling, resource planning, subcontractor procurement and evaluation, and manipulation and use of the technical data during remedial investigations and feasibility studies. This guidance is not intended to provide a detailed description of methodology and operation. The highly specialized nature of the subject geophysical methods requires inclusion of project-specific, site-specific, and subcontractor-specific information prior to development of detailed operating procedures, during both planning and execution.

The description focuses on methods and equipment that are readily available and typically applied; it is not intended to provide a complete discussion of the state of the art.

3.0 GLOSSARY

Magnetic Survey – A geophysical survey method that depends on detection of magnetic anomalies caused by the presence of buried ferromagnetic objects.

Magnetic Susceptibility – Property of a material corresponding to its ability to distort an applied magnetic field.

Magnetometer – A device used for precise and sensitive measurements of magnetic fields.

Magnetometry – The science of measuring variations in the earth's magnetic field.

Metal detection – A geophysical survey method that is based on electromagnetic coupling caused by underground conductive objects.

Vertical Gradiometer – A magnetometer equipped with two sensors that are vertically separated a fixed distance apart. It is best suited to map near surface features and is less susceptible to deep geologic features.

4.0 RESPONSIBILITIES

Project Manager – responsible for scoping the magnetic or metal detection surveys during development of the Work Plan with the help of the site geologist and site geophysicist.

Field Operations Leader (FOL) – responsible for overall management and coordination of the field effort.

Site Geophysicist – central role in determining the technique used for providing necessary data. Field work for these surveys is supervised by the site geophysicist, with support from geophysical technical

specialists and other personnel as needed. Data reduction and interpretation are performed by the site geophysicist or technical specialists.

5.0 PROCEDURES

5.1 Description of Methods

5.1.1 Theory and Principles of Operation

~~5.1.1.1 Magnetometry~~

~~Materials subject to a magnetic field will develop an induced magnetization, proportional to the applied magnetic field and the magnetic susceptibility of the material.~~

~~Induced magnetization in an object produces a local magnetic field which either reinforces (positive magnetic susceptibility) or reduces (negative susceptibility) the external applied field. The variations in an otherwise homogenous field caused by the presence of the object is called a magnetic anomaly, and observations of such anomalies can be used to infer the presence of magnetic objects.~~

~~Because there are numerous factors that affect magnetic fields there is no unique interpretation of a set of magnetometry data. Conversely, there is no unique magnetic anomaly produced by a particular kind of buried object. Factors that influence the response of a magnetometer to buried objects include the size, shape, depth, orientation, and magnetic susceptibility of the buried material. Various magnetometers are available such that many objects of interest at hazardous waste sites (particularly buried ferromagnetic materials such as drums, tanks, pipes and iron scrap) are detectable. While the location of ferromagnetic material can be detected to the precision of the survey, difficulties may be encountered in interpreting and attempting to identify the source of magnetic anomalies.~~

5.1.1.2 Metal Detection

When a radio frequency electromagnetic field generated by a transmitter coil encounters a highly conductive object such as metal (not necessarily ferromagnetic), alternating currents are induced in the object that, in turn, generate alternating secondary magnetic fields that are detected as alternating voltages by a receiver coil. The presence of the metal object effectively "couples" the transmitter and receiver coils, which otherwise are oriented so that little or no coupling exists. The principles of metal detector operation are very similar to those associated with electromagnetic induction instruments.

A number of factors influence the response of a metal detector. The receiver response increases with the size and surface area, and decreases with the depth of a buried object. Factors such as soil properties and object shape complicate detectability and interpretation. Certain shapes, such as elongated metal rods, are difficult to detect. Iron minerals and conductive fluids will affect the detector response in much the same manner as a target of interest. Generally, metal detectors show greater response to smaller nearby targets than to larger targets at greater depth, and the presence of widespread metallic debris at a site can interfere with attempts to detect buried drums and other objects.

5.1.2 General Applicability

5.1.2.1 Magnetic Surveys (Magnetometry)

Magnetometer and gradiometer surveys are useful in environmental and engineering projects that require a technique for mapping buried vertically oriented metallic pipe (e.g., locating a buried water well casing), or mapping stratigraphy or geologic structure in an igneous settings. Magnetometers are also a useful tool for mapping buried metallic debris, metallic utilities or metallic containers.

Magnetic surveys can more readily detect metallic masses than identify or characterize targets. Calculations of the mass or size of detected objects generally yield only approximate results.

Magnetic surveys may be impractical in areas where metal pipes, fences, railroad tracks, metal buildings, and other ferrous metal artifacts are abundant. However, proper selection of equipment and survey techniques can often alleviate some of these problems.

5.1.2.2 Metal Detectors

Metal detectors (MDs) can be used for locating buried metallic containers of various sizes; defining the boundaries of trenches containing metallic containers; locating buried metallic storage tanks; locating buried metallic pipes; avoiding buried utilities when drilling or trenching; or locating utility trenches which may provide a permeable pathway for contaminants.

The detection range of a MD is relatively short. Its sensitive areas are focused directly above and below the coil providing good definition of object location. Quart-sized metal objects can be detected at a distance of about 1 meter; objects the size of a 55 gallon drum can be detected up to 3 meters; and massive piles of metals can be detected at depths of 3 to 6 meters. Deeper objects are difficult to detect with an MD. Although most MDs are operated on foot, some can be vehicle-mounted if desired.

5.1.3 Instrumentation

5.1.3.1 Magnetometers

Three types of magnetometers, the fluxgate, proton precession, and the cesium vapor magnetometers, are commonly used at hazardous waste sites. The fluxgate magnetometer uses an iron core of high magnetic susceptibility as a sensor. The amount of coiled electrical current necessary to induce magnetic saturation of the rod is directly dependent upon, and thus measures, the strength of the ambient magnetic field. In a proton precession magnetometer a strong magnetic field is applied to a sensor filled with proton-rich fluid (e.g., kerosene) that realigns the protons. The field is then turned off and the frequency of the signal generated by the protons as they realign themselves ("precess") to the earth's magnetic field is dependent upon and measures the strength of the field at that point. The third common type of magnetometer is the cesium vapor (alkali-vapor) magnetometer. The cesium vapor magnetometer is capable of obtaining an order of magnitude greater sensitivity than the proton precession magnetometer. The cesium vapor magnetometer operates via a beam of polarized light from a cesium vapor lamp that is passed through a cell of cesium vapor. The atoms of the vapor become excited as they absorb greater amounts of the polarized light. The vapor in the cell eventually reaches an energy state that can no longer absorb the light and renders the cell transparent. A radio-frequency magnetic field causes the atoms of the vapor to shift back to an energy state that allows the vapor to again absorb the polarized light. The frequency required to return the vapor to an energy state that allows the cell to absorb light is a function of the ambient magnetic field. Some magnetometers, such as the fluxgate, are extremely

sensitive to orientation during measurement. In order to alleviate this problem, two sensors are connected by a rigid pole to form a "gradiometer" that measures only a certain directional component of the earth's magnetic field. These gradiometers are commonly used at hazardous waste sites.

The type of magnetometer best suited for a particular site investigation depends upon characteristics of that site and should be chosen by a person familiar with the different instruments available. Proton precession magnetometers, while very useful in many situations, will cease to function in an area with high magnetic gradients such as a junkyard or near a steel bridge.

Different instruments have different levels of sensitivity. Whereas in some cases, high sensitivity may be desired to detect deeply buried objects, in other instances, a low sensitivity instrument may be desired to reduce the effects of "noise" from nearby fences or cars. Furthermore, the size of the survey area and the resolution required will determine whether the magnetometer used is hand-held for stationary measurements or a vehicle-mounted continuous sensor model.

5.1.3.2 Metal Detectors (MDs)

Three general classes of metal detectors are commonly used in hazardous waste site studies: pipeline/cable locators, conventional "treasure hunter" detectors, and specialized detectors. The pipeline/cable detectors are commonly used by EPA field investigation teams. They do not respond to small objects like soda cans. Although most of the "treasure hunter" type detectors are used for locating coin-sized objects, some can be fitted with larger sensor coils suitable for detection of larger objects at greater depths. Some of these models also can operate under adverse soil conditions such as soils high iron content. Specialized detectors are also available to operate to greater depths, over a wide sweep area, operate continuously, cope with special field problems, or operate while vehicle-mounted. These special MDs require an experienced operator and are not commonly available.

5.2 Data Acquisition

5.2.1 Field Procedures

5.2.1.1 Magnetics

Magnetic measurements are generally made in a cross-grid pattern, or if a continuous sensor is used, in a series of parallel lines across the survey area. The desired resolution (reconnaissance or high density) and the size and depth of the objects sought, determines the spacing of measurement stations or survey lines. Because of the phenomenon of temporal magnetic drift, a magnetic survey must include a base station where magnetic measurements are made at regular intervals. A separate base station magnetometer is used to monitor fluctuations in the earth's magnetic field. The base station magnetometer is time synchronized with the mobile magnetometer and placed in an area of the site believed to be free of metals or other anthropogenic features. The base station magnetometer is configured to record one data point at a set time interval (e.g., every 5 seconds). At the completion of the survey, the base station magnetometer is interfaced with the mobile magnetometer and the total field magnetic data are automatically corrected for any observed diurnal drift.

Special care must be taken with handling of the magnetometer during use. The operator must not take measurements with the sensor near ferromagnetic objects such as belt buckles or steel-toed boots. The orientation of the magnetometer and its height from the ground must also be carefully controlled during operation. Recorded data must be annotated with station locations to allow construction of a site magnetic map.

5.2.1.2 Metal Detector

Surveys with metal detectors are similar in planning to those with magnetometers. A grid pattern of evenly spaced parallel lines is used. Desired resolution and the size of objects determine spacing. In some cases, elevating the MD a few feet off the ground may help to eliminate noise from small surface objects. An experienced operator is recommended. Recorded data must be annotated with station locations to allow construction of a site metal detection map.

5.2.2 Data Format

~~5.2.2.1 Magnetics~~

~~Most magnetometers are equipped with a solid state data logger that records the total field magnetic and/or the vertical gradient values, the survey line location, the survey station location and the time of the reading. The common units (SI) for total field magnetic data are Teslas (T). Magnetometers record total field data in units of nanoTeslas (nT). However, older texts may also refer to magnetic values as gammas (one gamma equals one nanoTesla). Vertical gradient data are commonly recorded in units of nanoTeslas per meter (nT/M).~~

5.2.2.2 Metal Detection

The data provided by a metal detector is less quantitative than that of a magnetometer. The MD signal strength may vary (depending on the instrument) with object depth, size, and shape, but this signal does not translate into a quantity such as field strength. It merely indicates the presence of a metal object. This on/off type of signal is useful because it can indicate the boundaries of a metal-bearing zone more clearly than some quantitative data such as magnetometer recordings.

5.3 Data Interpretation

~~**5.3.1 Magnetics**~~

~~5.3.1.1 Correction of Diurnal Variations~~

~~Diurnal drift is automatically corrected for by interfacing the mobile magnetometer with the base station magnetometer. However, all of the diurnal corrections should be checked to verify the values and insure against instrument malfunction.~~

~~5.3.1.2 Depth Estimates from Total Field~~

~~The width of a magnetic anomaly is proportional to the depth (or distance) of the source from the magnetometer sensor; the deeper the source, the broader the anomaly. This relationship is of primary importance in interpreting the results of a magnetic survey. The proportion between the width of an anomaly and the depth of the source is a function of the fall-off rate, or the variation of anomaly amplitude with distance(d). For a dipole, the total-field anomaly amplitude varies as $1/d^3$, and for a monopole as $1/d^2$. In actual practice, source orientation and other factors may result in fall-off rates from 1/d to $1/d^3$. The shape of the magnetic profile of an anomaly and knowledge of the source object help in selecting the proper fall-off rate for depth estimation. A range of depths determined from several fall-off rates may be the most appropriate way to present depth estimates.~~

In general the anomaly width is on the order of one to three times the depth of the source. Thus, for an anomaly with a width of 100 feet, the source is probably between 30 and 100 feet deep (or distant). Several methods, including the half-width rule and the slope technique, can be used to estimate source depths from total field profiles.

5.3.1.3 Half-Width Rule

The half-width ($x_{1/2}$) of an anomaly on a total field profile is the horizontal distance between the principal maximum (or minimum) of the anomaly (assumed to be over the center of the source) and the point where the total field value is exactly one-half of the principal maximum (Figure 3.2-1). A profile that is used for depth estimation by using the half-width rule should be oriented perpendicular to the long axis of the anomaly to give the narrowest profile. This rule is valid only for forms such as spheres, cylinders, and other simple shapes. For example, a single upright 55-gallon steel drum can be approximated as a vertical cylinder (monopole) and the depth (d) = $1.3\ x_{1/2}$. A buried trench filled with drums can be approximated by a horizontal cylinder, where $d = 2\ x_{1/2}$.

5.3.1.4 Slope Techniques

Depth of the source can be estimated using the slope of the anomaly at the inflection points of the profile. The horizontal extent (X_z) of the "straight" portion of the slope is determined as shown in Figure 3.2-1. The depth is then estimated by the equation,

$$d = KX_z \text{ where } 0.5 < K < 1.5$$

5.3.2 Metal Detection

Very little interpretation is necessary for metal detection surveys performed to provide qualitative data on the presence of metallic objects in the survey area, as a precursor to more detailed subsequent geophysical surveys. For these cases, the positive audible responses or meter deflections are recorded on site grid maps and no further processing or interpretation is made. More detailed metal detection surveys using strip-chart or magnetic tape recording are possible. Typically, data are plotted on site grid maps following computer processing. Corrections for nonlinearities and smoothing of the data to eliminate small-target responses can be accomplished.

5.4 Applications Management

5.4.1 Prerequisites

As described in Section 5.1.2, appropriate planning of magnetic and metal detection surveys requires at least a basic understanding of general site features and hydrogeologic characteristics, as well as the probable variability in conditions. The Work Plan should describe, in as much detail as possible, the known site conditions which may affect the measurements, and the objectives of proposed survey efforts. The type and degree of data interpretation and the desired format for data presentation should be specified if possible.

5.4.2 Work Planning and Scheduling

Magnetic and metal detection surveys may be performed concurrently with field investigations, in which case on-site interpretation of data may provide real-time guidance for well drilling activities. Ideally,

however, these geophysical surveys should be conducted in advance, allowing sufficient time for data interpretation and use of the results in planning other field exercises.

The time and effort required by these geophysical surveys vary greatly depending on the site-specific objectives and site conditions. Typically, 2 to 10 acres of metal detection can be performed by one person per day, depending on the complexity of the site. Additionally, 2 to 3 linear miles of magnetometer data can be collected by 1 person per day. Data reduction and interpretation will require at least an equivalent amount of time to the field work. Weather conditions, terrain, and obstructive site features cause considerable variability in these estimates.

5.5 Calibration

~~5.5.1 Magnetic Survey~~

Magnetometer readings should be compared regularly to readings of a reference base station magnetometer; this procedure is necessary if corrections are to be made for changes in the earth's magnetic field over time.

5.5.1.1 Daily Quality Control

All data sets should be accompanied by quality control data that indicates the level of quality of each individual data point. Periodically, replicate measurements should be made so that measurement precision can be established. This procedure also requires corrections for variations in the earth's magnetic field with time. Each data set should be referenced to the most recent calibrations. All data obtained prior to a calibration requiring significant changes in instrument controls are suspect, and the measurements should be repeated or otherwise validated. Data should be preliminarily reduced and plotted during the field program to determine the overall quality of the data and whether the survey results are consistent with the site conceptualization. Data points representing discontinuities in the curves should be validated by repetition and, if necessary, a fine grid of measurements made to determine whether the anomaly represents a site feature of interest, a spurious reading, or an obstructive interference.

The earth's magnetic field varies constantly due, primarily, to solar activity. These natural fluctuations must be accounted for and removed from the survey data. A second magnetometer will be used as a base station to measure and record these fluctuations. These data will subsequently be used to drift correct the survey magnetometer data. In addition, the U.S. Space Environmental Agency should be contacted daily to obtain the latest solar activity forecasts. Data acquisition will cease in the event of a magnetic storm. The phone number for the U.S. Space Environmental Agency is (303) 497-3171.

5.5.2 Metal Detection

5.5.2.1 Calibration

Metal detectors normally are not calibrated, and only relative response is of interest. Periodically, the sensitivity should be checked by nulling the instrument at a fixed location known to be free of metal, and adjusting the gain to provide a proper response over a known target.

5.5.2.2 Daily Quality Control

Metal detector data should be accompanied by sufficient quality control data to verify that the instrument was operating properly. Occasional repetitive measurements and a log of the sensitivity adjustments usually suffice for this purpose.

5.6 LIMITATIONS

Magnetometer data may be adversely affected by the presence of anthropogenic surface features such as buildings, fences, power lines, vehicles, reinforced concrete, and other metal objects. The magnetic response from these surface features can be much larger than that due to a single buried steel drum, and thus can mask the response of a drum. Magnetometer surveys should not be conducted in urban areas or areas where surface anthropogenic features are prevalent.

Data interpretation is not always straightforward with magnetic data. Metallic drums that are buried at the same depth but at different orientations can yield very dissimilar instrument responses. Data that are collected along survey lines oriented east-west can appear very different than data that are collected along survey lines oriented north-south (the preferred survey orientation).

6.0 REFERENCES

Good discussions of various geophysical survey techniques and applications are found in the following references:

Benson, R. C., R. A. Glaccum and M. R. Noel, 1982. Geophysical Techniques for Sensing Buried Wastes and Waste Migration, Technos, Inc., Miami, Florida, Contract No. 68-03-3050, U.S. EPA Environmental Monitoring Systems Laboratory, Las Vegas, Nevada.

Costello, R. L., 1980. Identification and Description of Geophysical Techniques, Report No. DRXTH-TE-CR-80084, U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, Maryland; Defense Technical Information System Number ADA 123939.

McKown, G. L., G. A. Sandness and G. W. Dawson, 1980. Detection and Identification of Buried Waste and Munitions, Proceedings of the 11th American Defense Preparedness Association Environmental Systems Symposium, Arlington, Virginia, 1980.

Ward, Stanley, H. 1990. Geotechnical and Environmental Geophysics, Society of Exploration Geophysicists. Tulsa, Oklahoma.

7.0 RECORDS

The following information will be recorded in the field logbook.

- Date
- Equipment operators
- Name and project number of site
- Position and instrument readings or responses if not recorded by a data logger
- Position-specific information
- Field sketches

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		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject GROUND-PENETRATING RADAR SURVEYS		Approved D. Senovich	

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1.0 PURPOSE

The purpose of this guideline is to provide a general description of, and technical management guidance on, the use of Ground-Penetrating Radar (GPR) Surveys.

2.0 SCOPE

This guideline provides a description of the principles of operation, instrumentation, applicability and implementability of standard GPR methods used during site investigations, to determine subsurface stratigraphic and other interfaces discernable by GPR. The document is intended to be used by the Project Manager (PM), Field Operations Leader (FOL), or site geophysicist to develop an understanding of each method sufficient to permit work planning, scheduling, and resource planning. This guidance is not intended to provide a detailed description of methodology and operation. The highly specialized nature of GPR surveys requires inclusion of project-specific, site-specific, and subcontractor-specific information prior to development of detailed operating plans, during both planning and execution.

3.0 GLOSSARY

Dielectric Constant. Property of a medium that determines reflection, absorption, and transmission characteristics of a radar signal; a measure of the ability of a material to store charge when an electric field is applied. Also known as permittivity.

Depth of Investigation. The depth at which an object of interest can be detected in a GPR survey.

Radar Trace. A display of reflected signal strength on a graph of lateral distance along the ground versus the radar signal travel time (corresponding to vertical distance of penetration).

Two-Way Travel Time. The time required for a radar signal to travel from the antenna to a target and return to the antenna. Travel time is a function of the depth of an object and the dielectric constant (permittivity) of the medium (soil, rock, etc.).

4.0 RESPONSIBILITIES

Project Manager - Responsible for scoping of the ground-penetrating radar surveys during development of the Work Plan, with input from the FOL, site geophysicist, and site geologist.

Field Operations Leader (FOL) - Responsible for overall management and coordination of the field work.

Site Geophysicist - As a specialist in this field, the site geophysicist plays a central role in determining the appropriateness of this technique for providing necessary data. Field work for these surveys is supervised by the site geophysicist, with support from geophysical technical specialists and other personnel (i.e., site geologist) as needed. Data reduction and interpretation are performed by the site geophysicist or technical specialist.

5.0 PROCEDURES

5.1 Description of Methods

5.1.1 Theory and Principles of Operation

Commercially-available GPR units operate on the principle of time-domain reflectometry, in which the difference in strength and the time delay between a transmitted electromagnetic pulse and its reflection from an object are measured. The time delay or two-way travel time, t, is directly related to the propagation velocity of the electromagnetic waves, v, and to the distance between the transmitter and the object, D, as follows:

$$t = \frac{2D}{v}$$

Because GPR is normally used at or near the surface of the ground, the distance D corresponds to the depth of buried target(s) that reflect the radar signals.

The strength of a radar signal is a complex function of the distance traveled through the medium and the dielectric constant, the magnetic permeability, and the electrical conductivity of the medium. Radar signals are attenuated rapidly in materials with high dielectric constants. This attenuation in subsurface media is a function of the mineralogy and the water content. Thus, materials such as dry sands and gravels are least absorptive of radar signals, whereas wet clays are highly absorptive. The absorptive properties of the medium limits the depth of investigation, e.g., the depth at which targets can be detected.

Radar antennas are available which operate at frequencies centered on 25-900 MHz. Whereas the higher frequencies are able to detect smaller targets, the penetration depth is roughly inversely proportional to frequency. Thus, any GPR survey requires an analysis of the trade-off between resolution and depth of penetration so that the optimal frequency can be selected.

The amplitude of a radar reflection is a function of the composition, size shape, and depth of the target and contrast between permittivity of target and permittivity of surrounding material. High amplitude reflections are from objects exhibiting large differences in dielectric constant from the surrounding medium, and are large in size compared to the radar signal wave length.

5.1.2 General Applicability

GPR signals are reflected from any interface which corresponds to an abrupt change in dielectric constant. Therefore, both metallic and nonmetallic objects (including voids) as well as changes in geologic structure can be detected by this method. Because of the higher frequencies used, target resolution is considerably improved over seismic or resistivity sounding methods. However, the high frequencies also result in strong attenuation of the signals, particularly in clay materials with high moisture content. At 100 MHz, the depth of investigation in clay soil with 20 percent moisture content is 3 feet or less, whereas in dry clay, or a sand with 20 percent moisture, the penetration depth can extend to approximately 30 feet.

GPR can be a powerful method for locating and mapping buried drums, wooden objects, foundations, non-containerized wastes, underground utilities, and many other artifacts (including historical artifacts) at a site. Depending on whether sufficient depth of investigation can be achieved, the method can also be used to map saturated zones and bedrock contours, and locate sinkholes or fracture systems.

A major limitation to applicability is the degree of subjective interpretation that is employed. The problem of noting a small signal disturbance in a sea of clutter can be overcome somewhat by simultaneous observation of a large number of parallel tracks.

5.1.3 Instrumentation

The standard array of GPR instrumentation consists of a transmitter/antenna unit which is pulled along the ground; a control unit, containing a power supply and signal processing circuitry which is connected to the antenna by a cable; and a laptop computer or analog tape recorder. The system can be vehicle-mounted, and the transmitter can be connected via radio link to the signal processing and recording equipment.

5.2 Data Acquisition

5.2.1 Field Procedures

GPR surveys are performed by establishing a grid of parallel survey lines across the site and moving the radar antenna along each of these lines. A suitable means must be provided for determining the location of the radar unit along each of the lines, and for documenting this information on the recording medium. Typical systems measure the time and velocity of antenna motion, or determine the position of the antenna by synchronization signals from the wheels or tracks of the vehicle used to tow the antenna.

To determine the depth of anomalies noted on radar traces, it is necessary to convert the travel time data that are actually recorded. The velocity of electromagnetic waves in the subsurface medium at the site is determined by excavation to observed targets and measuring their depths. The velocity should be determined at several points in the area of interest. Depths to targets can be estimated by using published values of dielectric constants for a range of different earthen materials and the following formula:

$$D = \frac{ct}{2\sqrt{\varepsilon_r}} = \frac{V_m t}{2} \qquad V_m = \frac{c}{\sqrt{\varepsilon_r}}$$

Where:

D = depth to target (feet)
t = two-way travel time (nanoseconds)
c = velocity of light (1 foot/nanosecond)
ε_r = dielectric constant (dimensionless)
V_m = velocity of electromagnetic waves

Two-way travel times can also be calculated by collecting GPR data over targets of known depth (e.g., trenches, buried pipes/culverts).

Specific procedures for data acquisition are as follows:

1. The time scale of the GPR unit shall be checked regularly for accuracy. This can be done either on or off the site by placing the GPR unit at a known distance from the ground, a wall, etc., and measuring the two-way travel time to that reflecting surface in the air. The velocity of electromagnetic waves in air is 1 foot per nanosecond (3×10^8 m/sec.). The following equation shall be used:

$$t = 2d/c$$

where:

t = two-way travel time from antenna to the surface, (nanoseconds)
d = distance of antenna to the surface, (feet)
c = velocity of light in air, (1 foot/nanosecond)

2. Prior to conducting a survey, a GPR traverse should be conducted over a buried object of known depth (if available). From the two-way travel time and the measured burial depth of the object, the average electromagnetic wave velocity (V) in soil can be calculated from the following equation:

$$V = 2d/t$$

3. A short GPR traverse shall be repeated twice daily over a known feature prior to and after conducting daily operations. Technical judgment shall be exercised to ensure that variations between repeat readings are a result of changing soil conditions rather than the electronics.

5.2.2 Data Format

Reflected radar signals are electronically processed and displayed as an intensity-modulated time spectrum, where the time corresponds to target depth as described above. The series of signals corresponding to the reflected pulses as the antenna moves along a path forms a three-dimensional data set containing distance of traverse, depth, and intensity information.

Typically, the data are recorded on a personal computer with distance displayed along the X-axis, time (depth) displayed along the Y-axis, and the intensity given by the degree of amplitude of the reflection. In a typical survey, a series of parallel tracks are traversed by the GPR, and the series of resulting oscillograph traces provides XYZ locational information and intensity of reflection from targets of interest.

Although much of the data obtained in a GPR survey is automatically recorded by the instrument, additional information to identify and interpret each trace should be recorded in a field notebook or on log sheets. At a minimum the data records should contain the following information:

1. Project name, number and location.
2. Company or organization.
3. Date and time of day.
4. Operator's name.
5. Line and trace designation (also recorded directly on the signal recording medium).
6. Equipment serial numbers.
7. Antenna frequency.
8. Direction and speed of antenna movement.
9. Weather and temperature.
10. Site map coordinates at the beginning and end of the trace.
11. Notes, remarks or comments.
12. Electromagnetic velocity in the subsurface medium at the nearest calibration point.

5.3 Data Interpretation

Except for those systems which provide extensive data processing, interpretation of anomalies in GPR traces require considerable subjective evaluation by a trained geophysicist. Extensive experience is essential to distinguish target reflections from inherent system noise and interferences. In many cases, the anomalies due to targets of interest are small compared to varying reflections from the antenna system, the ground surface, geologic perturbations, and other interferences. Similarly, an acceptable interpretation of target depth from travel time data requires a knowledge of geophysical characteristics across the site.

A radar antenna transmits a "cone," rather than a thin beam of electromagnetic energy, so that reflections are obtained from objects not directly below the antenna. As the antenna moves across the plane of an object, reflections are obtained for a considerable distance along the antenna path. The signal travel times will vary during this process, corresponding to the distance between the antenna and the object. A discrete spherical target would exhibit a hyperbolic reflection pattern on the radar trace, with the apex of the hyperbola corresponding to the location and depth of the object. Multiple or odd-shaped targets, or targets of considerable size (compared to the radar wavelength) will exhibit complex reflection patterns consisting of overlapping hyperbolas. Thus, a true "picture" of subsurface objects is not obtained, and experience is necessary to translate the complex tracings into information indicating the target depth, size, and shape.

5.4 Applications Management

5.4.1 Prerequisites

Appropriate planning of GPR surveys requires at least a basic understanding of the geophysical characteristics of the site. The type and structure of soils and geologic formations should be known. A description of the site should include the depth, size, shape, and type of potential targets to be detected, as well as obstructive site features such as rough or wet terrain and underground utilities and/or structures. The existence of, and depth to known buried objects should be listed and mapped. Other specifications include the degree of locational resolution desired, probable weather conditions during site activities, and the type and sophistication of software required for data interpretation and presentation.

5.4.2 Work Planning and Scheduling

If possible, GPR surveys should be performed concurrently with other geophysical surveys. Radar data complement information from other geophysical methods such as seismic refraction, magnetometry, and resistivity in arriving at an interpretation of subsurface geohydrologic features and location of buried waste materials. This information is important in locating and selecting the appropriate type of monitoring wells.

The time and effort required to perform GPR surveys vary greatly depending on the sophistication of the available equipment and the complexity of the site. Assuming a 2-person team, simple hand-operated radar systems can cover from 1/4 to 1/2 acre per day, proper documentation, and simple interpretation. Vehicle-mounted systems with automatic data recording and processing can cover from 2 to 5 acres per day. Sophisticated data processing, detailed interpretations, and high-quality displays require considerable computer usage and approximately twice the time required for the actual field survey.

The specific objectives of the GPR survey should be defined in the Work Plan and should include the following elements:

1. Type of survey (level of detail) to be accomplished, and area to be covered.
2. Type, depth, size, and composition of targets of interest (if known).
3. Locational accuracy required.
4. Schedule limitations.
5. Degree of sophistication required for data presentation and interpretation.
6. Specific deliverables required.

5.5 Quality Control (QC)

5.5.1 General

Because of the specialized nature of the method and the highly subjective interpretations needed to process the data, GPR surveys are subject to misapplication, erroneous interpretation, and collection of inadequate or incomplete data. This susceptibility to misuse requires that an adequate quality control program be established. Quality control aspects include the following :

- Insistence on a defined scope of work, specifications, and data validation procedures.
- No data point should be rejected from a data set without appropriate justification; field data sheets should contain all observed data and the conditions that could impact data validation.
- All field data should be recorded in permanent ink in a bound field logbook, and each page signed and dated by the operator. The original unaltered field logbook should be retained in the project file.
- In general, the objectives of geophysical surveys can be met by relative measurements across an area or with depth. Absolute calibration is therefore of lesser importance than precision of measurements. However, a properly calibrated instrument provides an added measure of data validity and permits correlation and comparison of the associated data with site features and hydrogeologic characteristics not evident at the time of the field effort.
- An evaluation should be made of background noise, interferences, and obstructions at a site. These items should be recorded in the field logbook. These real-time quality control procedures aid field personnel in correction of noise sources over which they have control, in validating suspected external sources, and in early detection of problems that may jeopardize the survey results.

5.5.2 Daily Quality Control

All radar traces and interpreted data sets should be accompanied by quality control data that indicate the level of quality of the data. Periodic replicate measurements should be made so that measurement precision can be established. Time and/or depth calibrations should be performed on a daily basis.

A calibration that yields significant changes in instrument parameters or travel time may indicate the need for repetition of data or increased density of travel time calibrations in the area of interest. Graphical data should be reviewed during the field activities to determine that data quality is adequate, and whether the survey results appear to be consistent with geophysical conceptual model of the site.

5.6 Potential Problems

A wide variety of problems may be encountered during performance of a GPR survey. Problems can be expected to arise in the following areas:

5.6.1 Planning and Execution

Rarely is a GPR survey accomplished exactly according to the original plan. Site features not previously specified and other variations can occur that force changes in the details of the approach. However, the objectives of the survey, the general methodology, the amount and quality of data required, and the degree of data interpretation requested should remain unchanged. Project work scopes should be written with some degree of latitude to allow a change in plans whenever justified.

5.6.2 Noise and Interferences

Measurements can be affected severely by natural and by man-made sources of interference. Sources of system noise that degrade the quality of radar traces include improper spacing of antennas above ground, improper cable placement, location of antennas too close to other system components, and faulty instrument operation. Because reflections are obtained from any objective with a dielectric constant differing from the surroundings, large masses or high density of buried or surface rocks, metal, debris, wet soil, or structures can mask targets of interest. Some antennas are not shielded on top, and similar interfering reflections will be obtained from overhead objects such as trees, power lines and buildings. The site personnel must recognize the limitations posed by these obstructions, and take steps to minimize these interferences.

Topographic and geologic features can also interfere with acquisition of high-quality target detection data. Small depressions in the ground surface, the presence of boulders, clay lenses and moist soil zones affect both the detectability of a target and determination of its depth from the travel time.

Sources of electromagnetic energy in the vicinity, such as radio or television transmitters, or navigational radar antennas can result in spurious signals in the radar traces. In some cases, these problems can be minimized by judicious selection of radar and/or data communications frequency, and by scheduling the surveys during periods of transmission inactivity.

5.6.3 Weather Conditions

Because water is a good absorber of radar signals, wet weather has a very serious effect on the ability to perform GPR surveys. Physical difficulties in executing a survey over wet terrain also may be expected. The field activities should be planned, if possible, during periods when dry weather is expected. Schedules for surveys should account for the probability that moist soil conditions will exist.

5.6.4 Technical Difficulties

Preventable difficulties include equipment malfunction, or misapplication, poor operator training, and lack of applications experience. Other difficulties may arise because the geophysical character of the site is not as initially conceptualized. The effect of these problems can be minimized by early recognition, using responsive and responsible technical management. Interim, real-time scrutiny of the data by the site geophysicist and management personnel is essential. The site geophysicist or geophysical subcontractor must be responsive regarding equipment replacement, repair, or changes in personnel. The Project Manager and the FOL should be cognizant of technical difficulties beyond the control of the field personnel, and should recognize the need to change plans, change performers, or cancel a survey, as appropriate.

6.0 REFERENCES

Good discussions of various geophysical survey techniques and applications are found in the following references:

1. Benson, Richard C., Robert A. Glaccum and Michael R. Noel, Geophysical Techniques for Sensing Buried Wastes and Waste Migration. U.S. EPA Environmental Monitoring Systems Laboratory, Las Vegas, Nevada.

2. Costello, Robert L., 1980, Identification and Description Geophysical Techniques, Report No. DRXTH-TE-CR-80084, U.S. Army Toxic and Hazardous Materials Agency, Aberdeen Proving Ground, Maryland; Defense Technical Information System Number ADA 123939.

3. McKown G. L., G. A. Sandness and G. W. Dawson, Detection and Identification of Buried Waste and Munitions, Proceedings of the 11th American Defense Preparedness Association Environmental Systems Symposium, Arlington, Virginia.

7.0 RECORDS

The following information will be recorded in the field logbook.

- Date
- Type of equipment
- Name and project number of site
- Site conditions that may affect data collection
- Depth and location information regarding known targets used for determining the wave velocity
- Additional items mentioned in Section 5.2.2

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Subject SITE RECONNAISSANCE		Approved D. Senovich	

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1.0 PURPOSE

The purpose of a site reconnaissance is to collect both general and technical information which will support the scoping, scheduling, implementing project activities, and writing reports for an environmental investigation. This procedure is not intended as a guide for Phase I investigations or for Environmental Baseline Survey activities.

2.0 SCOPE

This procedure is applicable to the performance of a site reconnaissance for initial site characterization. The steps necessary to develop and carry out a site reconnaissance are presented here. These steps include a list of equipment and items which may be needed, areas of special interest during field observations, and methods by which the field observation team can ensure that necessary and appropriate observations have been made.

3.0 GLOSSARY

Site reconnaissance. An onsite inspection program used to identify site-specific conditions that control scheduling, manpower, and affect costs. A site reconnaissance usually consists of visual observations and, often, the use of field monitoring instruments to identify potential health and safety threats and potential sampling locations for site evaluation during subsequent field investigations.

4.0 RESPONSIBILITIES

Field Operations Leader (FOL) is responsible for ensuring that the survey is carried out in sufficient detail. To accomplish this, the FOL must assign the proper personnel and equipment to characterize the site adequately, in accordance with the requirements defined in this procedure and best engineering practices. Other disciplines which may be applicable include (but are not limited to): Geology/Hydrogeology; Health and Safety; Ecological Specialists; and/or Engineering. In addition, the FOL is responsible for supervising equipment preparation, including necessary calibrations, and supervising field data collection and documentation in accordance with the methods described in all referenced standard operation procedures.

Project Manager is responsible for the following:

- Supervising the retrieval and examination of available, applicable information regarding the site.
- Obtaining appropriate program approvals and ensuring the preparation of a site Health and Safety plan for the site reconnaissance.
- Coordinating the field activities with the client and regulatory agencies, as applicable.

Field Personnel are primarily responsible for observing and documenting, either through written documentation or photographic evidence, the site reconnaissance. Field personnel will take direction from the FOL.

5.0 PROCEDURES

5.1 Equipment Items/Needed

Below is a list of items that may be useful when conducting a site reconnaissance. All, or a portion of these items may be required, depending upon the objective of the site reconnaissance.

- Health and Safety equipment and information as required by the Site Safety Officer.
- Maps (U.S.G.S. quadrangle, geologic maps, street and highway maps, and client facility maps).
- Geologic tools (compass, tape measure, hand level, camera, etc.).
- Physical monitoring equipment, if applicable (PID, Immunoassay Test Kits, etc.)
- Regional publications (U.S.G.S reports, water well surveys, U.S.D.A. soil conservation surveys, etc.).
- Site-specific publications by previous investigators (EPA aerial photographic analyses, remedial investigation reports, data on waste disposal practices, boring logs, etc.).
- Marking items (ink markers, surveyor's flagging, spray paint, pin flags, wooden stakes).
- Field notebooks.
- Local telephone book with yellow pages (for obtaining utilities, site trailer, living accommodations, etc.).

Sufficient time will be required in order to obtain some of the aforementioned material. In general, most publications can be obtained in time to be used in the site reconnaissance if ordered approximately 2 weeks before the actual site visit takes place.

5.2 Observations

A site reconnaissance usually requires one to two days, however, additional time may be needed depending upon the objective, site size, etc. The following observations, when applicable, should be documented either on a site map, field notebook, or photographed.

- General Site Access. It should be noted whether site roads provide access to all proposed work locations, or if it will be necessary to prepare access roads with either a backhoe, dozer, chain saws, etc., in order to get drill rigs, excavators, or other work vehicles to specific locations. If temporary driveways must be constructed from existing public roads, regulatory permits may be required. Military facilities may have specific security requirements which require detailed clearance procedures.
- Location of the Command Post or Site Trailer and Sanitary Facilities. The ideal location for the site trailer and sanitary facilities is a level area, within an uncontaminated zone, and centralized in order to provide easy access to work areas on the site. However, certain utility companies may require that the site trailer be placed within a specified radius (usually 100 feet), of the nearest utility pole. Contact the necessary utility companies and inquire about the requirements regarding service before conducting the site reconnaissance. Information that may be required by the utility companies is: type of electric service needed (inquire with trailer vendor for this information); and utility pole number of interest (pole numbers are usually stamped on a brass plate on the pole).

- Potable Water Sources. Local fire departments may allow access to fire hydrants. Private water delivery companies may also be available in the area.

- Sources of Possible Contamination. Drums, tanks, sludge areas, areas of stressed vegetation, fill areas, and leachate seeps may indicate where sources of contamination exist. Filler pipes protruding from the ground surface may indicate the presence of underground storage tanks. Areas where the original ground surface has been reworked may be contaminated fill areas that have since been buried and covered with natural material. Previous environmental investigations may also identify source areas.

- Location of Decon Areas and Storage/Disposal Areas for Equipment and Wastes Generated by Field Activities.

- Locations of Surface Water Bodies. The locations of surface water bodies, both man-made and natural, and their relation to topographic highs may give an indication of the groundwater flow direction in the area (groundwater flow typically follows topography with the topographic highs serving as groundwater recharge areas, and the surface waters at topographic lows serve as groundwater discharge areas). Visible signs of contamination, the existence of aquatic life, flow rates, and approximate levels should also be observed and noted. Check if the surface water bodies could potentially be impacted by field activities. If so, appropriate sedimentation and erosion controls will be required.

- Existing Wells. Existing monitoring wells, or domestic wells within the site and off site, should be noted on a map, and access checked to see if the wells can be used for data collection.

- Outcrops. Outcrops can be useful in providing hydrogeologic data (lithologic description, strike and dip information, fracture and joint system analysis, identification of moist zones, etc.) Outcrops may occur naturally or be a part of a man-made feature such as a road-cut.

- Lineaments. A lineament is a straight lengthy feature on the earth's surface which is expressed topographically as a line of depression. Stream beds, vegetation patterns or soil characteristics may be aligned or controlled by this feature. Lineaments are due in some cases to the presence of intense jointing or faults beneath the ground surface. Groundwater in the bedrock may follow lineaments. Lineaments should be noted on site maps and described in the field notebooks.

- Bench or Property Markers. Benchmarks or property markers should be marked with paint or surveyor's flagging if encountered during a site reconnaissance. Surveyors may need to use these markers as a reference point when surveying. Benchmarks are typically a brass plate secured in concrete in the ground with numbering on the top. Property markers can range from a stake driven into the ground to a rock protruding from the ground surface. Facility contacts may also be aware of local benchmarks used during the course of other environmental or public work projects.

- Metal Cultural Effects. Overhead power lines, railroad tracks, junk automobiles, fences, etc. will greatly affect certain geophysical surveys. These features should be noted while conducting a site reconnaissance.

6.0 RECORDS

The data collected during a site reconnaissance may have to be compiled into a trip report when returning from the field. This trip report can then be distributed to the project team. A site reconnaissance checklist is located in Attachment A which can be copied and used while conducting the site reconnaissance.

ATTACHMENT A

SITE RECONNAISSANCE CHECKLIST

SITE SKETCH

Include the following as appropriate:

- Site Name
- Site location
- Site Boundaries
- Entrance locations
- Access Roads and Security Requirements
- Disposal locations
- Storage areas
- Office areas
- Well locations
- Treatment facility locations
- Surface drainage, outcrops, general topography descriptions
- Cultural interferences

CHEMICAL STORAGE FACILITIES DESCRIPTION

- Storage tanks - numbers, volumes, condition, contents, etc.
- Drums - number, conditions, labeling, etc.
- Lagoons and surface pits - number, size, use of liner, contents, etc.

TREATMENT SYSTEMS

Note the presence of any treatment systems. These can be difficult to evaluate visually. One should appraise general appearance, maintenance and visual integrity; ask operators for any monitoring records; note presence of odors; and visually characterize any effluents or residues. Describe type of wastes and volumes treated.

- Incinerators
- Flocculation/filtration
- Chemical/physical treatment
- Biological treatment
- Volume reduction
- Waste recycling
- Compositing
- Other

ATTACHMENT A
SITE RECONNAISSANCE CHECKLIST
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DISPOSAL FACILITIES

Note the presence and use of any of the following operations. Include a description of the size, use of liners, soil type, and the presence of leachate. Provide a description of management practices. Interview site workers if possible. Describe waste types.

- Landfills
- Land forms
- Open dump
- Surface impoundment
- Underground injection
- Incineration

Also, records for disposal of concentrated/containerized waste should be reviewed.

HAZARDOUS SUBSTANCE CHARACTERISTICS

Ask facility contacts for manifests, inventories, or monitoring reports. Note markings on containers.

- Chemical identities
- Quantities
- Hazard characteristics (toxic, explosive, flammable, etc.)
- Container markings
- Monitoring data, other analytical data
- Physical state (liquid, solid, gas, sludge)

CHEMICAL PROCESS INFORMATION

- Manufacturing processes and chemicals
- Off-specification or by-product disposal processes
- Housekeeping practices
- Locations of Plant Operations

ATTACHMENT A
SITE RECONNAISSANCE CHECKLIST
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HYDROGEOLOGIC ASSESSMENT

Look for situations that promote hazardous substance migration, i.e., porous soils, fractured bedrock formations, shallow water table and karst features.

- Soil type
- Surface water features
- Surface drainage pattern
- Outcrop studies
- Water wells (use, water depth, and construction details)
- Erosion potential
- Flooding potential
- Climatology

IDENTIFICATION OF SENSITIVE RECEPTORS

- Number and locations of private homes
- Public buildings including tenant usage
- Areas of dead or dying vegetation or animals
- Presence of sensitive ecosystems (wetlands, tidal marshes, etc.)
- Other public use areas (roads, parks, etc.)
- Natural areas

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Subject EVALUATION OF EXISTING MONITORING WELLS AND WATER LEVEL MEASUREMENT		Approved D. Senovich	

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1.0 PURPOSE

The purpose of this procedure is to provide reference information regarding the proper methods for evaluating the physical condition and project utility of existing monitoring wells and determining water levels.

2.0 SCOPE

The procedures described herein are applicable to all existing monitoring wells and, for the most part, are independent of construction materials and methods.

3.0 GLOSSARY

Hydraulic Head - The height to which water will rise in a well.

Water Table - A surface in an unconfined aquifer where groundwater pressure is equal to atmospheric pressure (i.e., the pressure head is zero).

4.0 RESPONSIBILITIES

Site Geologist/Hydrogeologist - Has overall responsibility for the evaluation of existing wells, obtaining water level measurements and developing groundwater contour maps. The site geologist/hydrogeologist (in concurrence with the Project Manager) shall specify the reference point from which water levels are measured (usually a specific point on the upper edge of the inner well casing), the number and location of data points which shall be used for constructing a contour map, and how many complete sets of water levels are required to adequately define groundwater flow directions (e.g., if there are seasonal variations).

Field Personnel - Must have a basic familiarity with the equipment and procedures involved in obtaining water levels and must be aware of any project-specific requirements or objectives.

5.0 PROCEDURES

Accurate, valid and useful groundwater monitoring requires that four important conditions be met:

- Proper characterization of site hydrogeology.
- Proper design of the groundwater monitoring program, including adequate numbers of wells installed at appropriate locations and depths.
- Satisfactory methods of groundwater sampling and analysis to meet the project data quality objectives (DQOs).
- The assurance that specific monitoring well samples are representative of water quality conditions in the monitored interval.

To insure that these conditions are met, adequate descriptions of subsurface geology, well construction methods and well testing results must be available. The following steps will help to insure that the required data are available to permit an evaluation of the utility of existing monitoring wells for collecting additional samples.

5.1 Preliminary Evaluation

A necessary first step in evaluating existing monitoring well data is the study and review of the original work plan for monitoring well installation (if available). This helps to familiarize the site geologist/hydrogeologist with site-specific condition, and will promote an understanding of the original purpose of the monitoring wells.

The next step of the evaluation should involve a review of all available information concerning borehole drilling and well construction. This will allow interpretation of groundwater flow conditions and area geology, and will help to establish consistency between hydraulic properties of the well and physical features of the well or formation. The physical features which should be identified and detailed, if available, include:

- The well identification number, permit number and location by referenced coordinates, the distance from prominent site features, or the location of the well on a map.
- The installation dates, drilling methods, well development methods, past sampling dates, and drilling contractors.
- The depth to bedrock -- where rock cores were not taken, auger refusal, drive casing refusal or penetration test results (blow counts for split-barrel sampling) may be used to estimate bedrock interface.
- The soil profile and stratigraphy.
- The borehole depth and diameter.
- The elevation of the top of the protective casing, the top of the well riser, and the ground surface.
- The total depth of the well.
- The type of well materials, screen type, slot size, and length, and the elevation/depths of the screen, interval, and/or monitored interval.
- The elevation/depths of the tops and bottom of the filter pack and well seals and the type and size.

5.2 Field Inspection

During the onsite inspection of existing monitoring wells, features to be noted include:

- The condition of the protective casing, cap and lock.
- The condition of the cement seal surrounding the protective casing.
- The presence of depressions or standing water around the casing.
- The presence of and condition of dedicated sampling equipment.
- The presence of a survey mark on the inner well casing.

If the protective casing, cap and lock have been damaged or the cement collar appears deteriorated, or if there are any depressions around the well casing capable of holding water, surface water may have infiltrated into the well. This may invalidate previous sampling results unless the time when leakage started can be precisely determined.

The routine physical inspection must be followed by a more detailed investigation to identify other potential routes of contamination or sampling equipment malfunction. Any of these occurrences may invalidate

previously-collected water quality data. If the monitoring well is to be used in the future, considerations shown in the steps described above should be rectified to rehabilitate the well.

After disconnecting any wires, cables or electrical sources, remove the lock and open the cap. Check for the presence of organic vapors with a photoionization detector (PID) or flame-ionization detector (FID) to determine the appropriate worker safety level. The following information should be noted:

- Cap function.
- Physical characteristics and composition of the inner casing or riser, including inner diameter and annular space.
- Presence of grout between the riser and outer protective casing and the existence of drain holes in the protective casing.
- Presence of a riser cap, method of attachment to casing, and venting of the riser.
- Presence of dedicated sampling equipment; if possible, remove such equipment and inspect size, materials of construction and condition.

The final step of the field inspection is to confirm previous hydraulic or physical property data and to obtain data not previously available. This includes the determination of static water levels, total well depth and well obstruction. This may be accomplished using a weighted tape measure which can also be used to check for sediment (the weight will advance slowly if sediment is present, and the presence of sediment on the weight upon removal should be noted). If sediment is present and/or the well has not been sampled in 12 or more months, it should be redeveloped before sampling.

Lastly, as a final step, the location, condition and expected water quality of the wells should be reviewed in light of their usefulness for the intended purpose of the investigation.

See Attachment A, Monitoring Well Inspection Sheet.

5.3 Water Level (Hydraulic Head) Measurements

5.3.1 General

Groundwater level measurements can be made in monitoring wells, private or public water wells, piezometers, open boreholes, or test pits (after stabilization). Groundwater measurements should generally not be made in boreholes with drilling rods or auger flights present. If groundwater sampling activities are to occur, groundwater level measurements shall take place prior to well purging or sampling.

All groundwater level measurements shall be made to the nearest 0.01 foot, and recorded in the site geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet (Attachment B), along with the date and time of the reading. The total depth of the well shall be measured and recorded, if not already known. Weather changes that occur over the period of time during which water levels are being taken, such as precipitation and barometric pressure changes, should be noted.

In measuring groundwater levels, there shall be a clearly-established reference point of known elevation, which is normally identified by a mark on the upper edge of the inner well casing. To be useful, the reference point should be tied in with an established USGS benchmark or other properly surveyed elevation datum. An arbitrary datum could be used for an isolated group of wells, if necessary.

Cascading water within a borehole or steel well casings can cause false readings with some types of sounding devices (chalked line, electrical). Oil layers may also cause problems in determining the true water level in a well. Special devices (interface probes) are available for measuring the thickness of oil layers and true depth to groundwater, if required.

Water level readings shall be taken regularly, as required by the site geologist/hydrogeologist. Monitoring wells or open-cased boreholes that are subject to tidal fluctuations should be read in conjunction with a tidal chart (or preferably in conjunction with readings of a tide staff or tide level recorder installed in the adjacent water body); the frequency of such readings shall be established by the site hydrogeologist. All water level measurements at a site used to develop a groundwater contour map shall be made in the shortest practical time to minimize affects due to weather changes.

5.3.2 Water Level Measuring Techniques

There are several methods for determining standing or changing water levels in boreholes and monitoring wells. Certain methods have particular advantages and disadvantages depending upon well conditions. A general description of these methods is presented, along with a listing of various advantages and disadvantages of each technique. An effective technique shall be selected for the particular site conditions by the site geologist/hydrogeologist.

In most instances, preparation of accurate potentiometric surface maps require that static water level measurements be obtained to a precision of 0.01 feet. To obtain such measurements in individual accessible wells, electrical water level indicator methods have been found to be best, and thus should be utilized. Other, less precise methods, such as the popper or bell sound, or bailer line methods, should be avoided. When a large number of (or continuous) readings are required, time-consuming individual readings are not usually feasible. In such cases, it is best to use a pressure transducer.

5.3.3 Methods

Water levels can be measured by several different techniques, but the same steps shall be followed in each case. The proper sequence is as follows:

1. Check operation of recording equipment above ground. Prior to opening the well, don personal protective equipment, as required. Never remove an air-tight lock (such as a J-plug) with your face over the well. Pressure changes within the well may explosively force the cap off once loosened.

2. Record all information specified below in the geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet (Attachment B):

    - Well number.
    - Water level (to the nearest 0.01 foot). Water levels shall be taken from the surveyed reference mark on the top edge of the inner well casing. If the J-plug was on the well very tightly, it may take several minutes for the water level to stabilize.
    - Time and day of the measurement.
    - Thickness of free product if present.

Water level measuring devices with permanently marked intervals shall be used. The devices shall be free of kinks or folds which will affect the ability of the equipment to hang straight in the well pipe.

5.3.4 Water Level Measuring Devices

Electric Water Level Indicators

These are the most commonly used devices and consist of a spool of small-diameter cable and a weighted probe attached to the end. When the probe comes in contact with the water, an electrical circuit is closed and a meter, light, and/or buzzer attached to the spool will signal the contact.

There are a number of commercial electric sounders available, none of which is entirely reliable under all conditions likely to occur in a contaminated monitoring well. In conditions where there is oil on the water, groundwater with high specific conductance, water cascading into the well, steel well casing, or a turbulent water surface in the well, measuring with an electric sounder may be difficult.

For accurate readings, the probe shall be lowered slowly into the well adjacent to the survey mark on the inner well casing. The electric tape is read (to the nearest 0.01 ft.) at the measuring point and recorded where contact with the water surface was indicated.

~~**Popper or Bell Sounder**~~

~~A bell- or cup-shaped weight that is hollow on the bottom is attached to a measuring tape and lowered into the well. A "plopping" or "popping" sound is made when the weight strikes the surface of the water. An accurate reading can be determined by lifting and lowering the weight in short strokes, and reading the tape when the weight strikes the water. This method is not sufficiently accurate to obtain water levels to 0.01 feet, and thus is more appropriate for obtaining only approximate water levels quickly.~~

~~**Pressure Transducer**~~

~~Pressure transducers can be lowered into a well or borehole to measure the pressure of water and therefore the water elevation above the transducer. The transducer is wired into a recorder at the surface to record changes in water level with time. The recorder digitizes the information and can provide a printout or transfer the information to a computer for evaluation (using a well drawdown/recovery model). The pressure transducer should be initially calibrated with another water level measurement technique to ensure accuracy. This technique is very useful for hydraulic conductivity testing in highly permeable material where repeated, accurate water level measurements are required in a very short period of time. A sensitive transducer element is required to measure water levels to 0.01 foot accuracy.~~

~~**Borehole Geophysics**~~

~~Approximate water levels can be determined during geophysical logging of the borehole (although this is not the primary purpose for geophysical logging and such logging is not cost effective if used only for this purpose). Several logging techniques will indicate water level. Commonly used logs which will indicate saturated/unsaturated conditions include the spontaneous potential (SP) log and the neutron log.~~

5.3.5 Data Recording

Water level measurements, time, data, and weather conditions shall be recorded in the geologist/hydrogeologist's field notebook or on the Groundwater Level Measurement Sheet. All water level measurements shall be measured from a known reference point. The reference point is generally a marked point on the upper edge of the inner well casing that has been surveyed for an elevation. The exact reference point shall be marked with permanent ink on the casing since the top of the casing may not be entirely level. It is important to note changes in weather conditions because changes in the barometric pressure may affect the water level within the well.

5.3.6 Specific Quality Control Procedures for Water Level Measuring Devices

All groundwater level measurement devices must be cleaned before and after each use to prevent cross contamination of wells. Manufacturer's instructions for cleaning the device shall be strictly followed. Some devices used to measure groundwater levels may need to be calibrated. These devices shall be calibrated to 0.01 foot accuracy and any adjustments/corrections shall be recorded in the field logbook/notebook. After the corrections/adjustments are made to the measuring device and entered in the field logbook/notebook, the corrected readings shall be entered onto the Groundwater Level Measurement Sheet (Attachment B). Elevations will be entered on the sheet when they become available.

5.4 Equipment Decontamination

Equipment used for water level measurements provide a mechanism for potentially cross contaminating wells. Therefore, all portions of a device which project down the well casing must be decontaminated prior to advancing to the next well. Decontamination procedures vary based on the project objectives but must be defined prior to conducting any field activities including the collection of water level data. Consult the project planning documents and SA-7.1 Decontamination of Field Equipment.

5.5 Health and Safety Considerations

Groundwater contaminated by volatile organic compounds may release toxic vapors into the air space inside the well pipe. The release of this air when the well is initially opened is a health/safety hazard which must be considered. Initial monitoring of the well headspace and breathing zone concentrations using a PID or FID shall be performed to determine required levels of protection. Under certain conditions, air-tight well caps may explosively fly off the well when the pressure is relieved. Never stand directly over a well when uncapping it.

6.0 RECORDS

A record of all field procedures, tests and observations must be recorded in the site logbook or designated field notebook. Entries in the log/notebook should include the individuals participating in the field effort, and the date and time. The use of annotated sketches may help to supplement the evaluation.

ATTACHMENT A
MONITORING WELL INSPECTION SHEET

Monitoring Well Inspection Sheet

Project Name: _____ Date: _____

Location: _____ Time: _____

Tidally influenced: Y / N Personnel: _____

Field Measurements				
Well ID	PID Reading PPM	Depth to Water *	Total Depth *	Flush Mt./ Stick-up

Well Construction Details (Taken from construction logs)		
Total Depth *	Ground Elev.	Top/Btm Screen *

Check List:

Riser Pipe Material:
Riser Notched for Surveyors:
Well ID Tag in-place:
Well security:
Photo taken:

Condition of Well:

Protective Case:
Riser:
Well Pad:
Other:

Presence/Evidence of:

Standing Water Around Well:
Existing Sampling Equipment:
Sediment build-up in Well Btm:

Comments:

** = Measurements are from the top of the inner case to the nearest 0.01'*

ATTACHMENT B
GROUNDWATER LEVEL MESUREMENT SHEET

Tetra Tech NUS, Inc.

GROUNDWATER LEVEL MEASUREMENT SHEET

Project Name: _____ **Project No.:** _____
Location: _____ **Personnel:** _____
Weather Conditions: _____ **Measuring Device:** _____
Tidally Influenced: Yes ___ No ___ **Remarks:** _____

Well or Piezometer Number	Date	Time	Elevation of Reference Point (feet)*	Water Level Indicator Reading (feet)*	Thickness of Free Product (feet)	Comments

* All measurements to the nearest 0.01 foot, from surveyed mark (unless noted)

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		Prepared Earth Sciences Department	
Subject SOIL AND ROCK DRILLING METHODS		Approved D. Senovich	

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FIGURE

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1.0 PURPOSE

The purpose of this procedure is to describe the methods and equipment necessary to perform soil and rock borings and identify the equipment, sequence of events, and appropriate methods necessary to obtain soil, both surface and subsurface, and rock samples during field sampling activities.

2.0 SCOPE

This guideline addresses most of the accepted and standard drilling techniques, their benefits, and drawbacks. It should be used generally to determine what type of drilling techniques would be most successful depending on site-specific geologic conditions and the type of sampling required.

The sampling methods described within this procedure are applicable to collecting surface and subsurface soil samples, and obtaining rock core samples for lithologic and hydrogeologic evaluation, excavation/foundation design, remedial alternative design and related civil engineering purposes.

3.0 GLOSSARY

Rock Coring - A method in which a continuous solid cylindrical sample of rock or compact rock-like soil is obtained by the use of a double tube core barrel that is equipped with an appropriate diamond-studded drill bit which is advanced with a hydraulic rotary drilling machine.

Wire-Line Coring - As an alternative to conventional coring, this technique is valuable in deep hole drilling, since this method eliminates trips in and out of the hole with the coring equipment. With this technique, the core barrel becomes an integral part of the drill rod string. The drill rod serves as both a coring device and casing.

4.0 RESPONSIBILITIES

Project Manager - In consultation with the project geologist, the Project Manager is responsible for evaluating the drilling requirements for the site and specifying drilling techniques that will be successful given the study objectives and the known or suspected geologic conditions at the site. The Project Manager also determines the disposal methods for products generated by drilling, such as drill cuttings and well development water, as well as any specialized supplies or logistical support required for the drilling operations.

Field Operations Leader (FOL) - The FOL is responsible for the overall supervision and scheduling of drilling activities, and is strongly supported by the project geologist.

Project Geologist - The project geologist is responsible for ensuring that standard and approved drilling procedures are followed. The geologist will generate a detailed boring log for each test hole. This log shall include a description of materials, samples, method of sampling, blow counts, and other pertinent drilling and testing information that may be obtained during drilling (see SOPs SA-6.3 and GH-1.5). Often this position for inspecting the drilling operations may be filled by other geotechnical personnel, such as soils and foundation engineers, civil engineers, etc.

Determination of the exact location for borings is the responsibility of the site geologist. The final location for drilling must be properly documented on the boring log. The general area in which the borings are to be located will be shown on a site map included in the Work Plan and/or Sampling and Analysis Plan.

Drilling Subcontractor - Operates under the supervision of the FOL. Responsible for obtaining all drilling permits and clearances, and supplying all services (including labor), equipment and material required to perform the drilling, testing, and well installation program, as well as maintenance and quality control of such required equipment except as stated in signed and approved subcontracts.

The driller must report any major technical or analytical problems encountered in the field to the FOL within 24 hours of determination, and must provide advance written notification of any changes in field procedures, describing and justifying such changes. No such changes shall be made unless requested and authorized in writing by the FOL (with the concurrence of the Project Manager). Depending on the subcontract, the Project Manager may need to obtain written authorization from appropriate administrative personnel before approving any changes.

The drilling subcontractor is responsible for following decontamination procedures specified in the project plan documents. Upon completion of the work, the driller is responsible for demobilizing all equipment, cleaning up any materials deposited on site during drilling operations, and properly backfilling any open borings.

5.0 PROCEDURES

5.1 General

The purpose of drilling boreholes is:

- To determine the type, thickness, and certain physical and chemical properties of the soil, water and rock strata which underlie the site.
- To install monitoring wells or piezometers.

All drilling and sampling equipment will be cleaned between samples and borings using appropriate decontamination procedures as outlined in SOP SA-7.1. Unless otherwise specified, it is generally advisable to drill borings at "clean" locations first, and at the most contaminated locations last, to reduce the risk of spreading contamination between locations. All borings must be logged by the site geologist as they proceed (see SOPs SA-6.3 and GH-1.5). Situations where logging would not be required would include installation of multiple well points within a small area, or a "second attempt" boring adjacent to a boring that could not be continued through resistant material. In the latter case, the boring log can be resumed 5 feet above the depth at which the initial boring was abandoned, although the site geologist should still confirm that the stratigraphy at the redrilled location conforms essentially with that encountered at the original location. If significant differences are seen, each hole should be logged separately.

5.2 Drilling Methods

The selected drilling methods described below apply to drilling in subsurface materials, including, but not limited to, sand, gravel, clay, silt, cobbles, boulders, rock and man-made fill. Drilling methods should be selected after studying the site geology and terrain, the waste conditions at the site, and reviewing the purpose of drilling and the overall subsurface investigation program proposed for the site. The full range of different drilling methods applicable to the proposed program should be identified with final selection based on relative cost, availability, time constraints, and how well each method meets the sampling and testing requirements of the individual drilling program.

5.2.1 Continuous-Flight Hollow-Stem Auger Drilling

This method of drilling consists of rotating augers with a hollow stem into the ground. Cuttings are brought to the surface by the rotating action of the auger. This method is relatively quick and inexpensive. Advantages of this type of drilling include:

- Samples can be obtained without pulling the augers out of the hole. However, this is a poor method for obtaining grab samples from thin, discrete formations because of mixing of soils which occurs as the material is brought to the surface. Sampling of such formations requires the use of split-barrel or thin-wall tube samplers advanced through the hollow core of the auger.
- No drilling fluids are required.
- A well can be installed inside the auger stem and backfilled as the augers are withdrawn.

Disadvantages and limitations of this method of drilling include:

- Augering can only be done in unconsolidated materials.
- The inside diameter of hollow stem augers used for well installation should be at least 4 inches greater than the well casing. Use of such large-diameter hollow-stem augers is more expensive than the use of small-diameter augers in boreholes not used for well installation. Furthermore, the density of unconsolidated materials and depths become more of a limiting factor. More friction is produced with the larger diameter auger and subsequently greater torque is needed to advance the boring.
- The maximum effective depth for drilling is 150 feet or less, depending on site conditions and the size of augers used.
- In augering through clean sand formations below the water table, the sand will tend to flow into the hollow stem when the plug is removed for soil sampling or well installation. If the condition of "running" or "flowing" sands is persistent at a site, an alternative method of drilling is recommended, in particular for wells or boreholes deeper than 25 feet.

Hollow-stem auger drilling is the preferred method of drilling. Most alternative methods require the introduction of water or mud downhole (air rotary is the exception) to maintain the open borehole. With these other methods, great care must be taken to ensure that the method does not interfere with the collection of a representative sample (which may be the prime objective of the borehole construction). With this in mind, the preferred order of choice of drilling method after hollow-stem augering (HSA) is:

- Cable tool
- Casing drive (air)
- Air rotary
- Mud rotary
- Rotosonic
- Drive and wash
- Jetting

However, the use of any method will also depend on efficiency and cost-effectiveness. In many cases, mud rotary is the only feasible alternative to hollow-stem augering. Thus, mud rotary drilling is generally acceptable as a first substitute for HSA.

The procedures for sampling soils through holes drilled by hollow-stem auger shall conform with the applicable ASTM Standards: D1587-83 and D1586-84. The guidelines established in SOP SA-1.3 shall

also be followed. The hollow-stem auger may be advanced by any power-operated drilling machine having sufficient torque and ram range to rotate and force the auger to the desired depth. The machine must, however, be equipped with the accessory equipment needed to perform required sampling, or rock coring.

The hollow-stem auger may be used without the plug when boring for geotechnical examination or for well installation. However, when drilling below the water table, specially designed plugs which allow passage of formation water but not solid material shall be used (see Reference 1 of this guideline). This drilling configuration method also prevents blow back and plugging of the auger when the plug is removed for sampling.

Alternately, it may be necessary to keep the hollow stem full of water, at least to the level of the water table, to prevent blowback and plugging of the auger. If water is added to the hole, it must be sampled and analyzed to determine if it is free from contaminants prior to use. In addition, the amount of water introduced, the amount recovered upon attainment of depth, and the amount of water extracted during well development must be carefully logged in order to ensure that a representative sample of the formation water can be obtained. Well development should occur as soon after well completion as practicable (see SOP GH-2.8 for well development procedures). If gravelly or hard material is encountered which prevents advancing the auger to the desired depth, augering should be halted and either driven casing or hydraulic rotary methods should be attempted. If the depth to the bedrock/soil interface and bedrock lithology must be determined, then a 5-foot confirmatory core run should be conducted (see Section 5.2.9).

At the option of the Field Operations Leader (in communication with the Project Manager), when resistant materials prevent the advancement of the auger, a new boring can be attempted. The original boring must be properly backfilled and the new boring started a short distance away at a location determined by the site geologist. If multiple water bearing strata were encountered, the original boring must be grouted. In some formations, it may be prudent to also grout borings which penetrate only the water table aquifer, since loose soil backfill in the boring may still provide a preferred pathway for surface liquids to reach the water table. Backfilling requirements may also be driven by state or local regulations.

5.2.2 ~~Continuous-Flight Solid-Stem Auger Drilling~~

~~This drilling method is similar to hollow-stem augering. Practical application of this method is severely restricted compared to use of hollow-stem augers. Split-barrel (split-spoon) sampling cannot be performed without pulling the augers out, which may allow the hole to collapse. The continuous flight solid-stem auger drilling method is therefore very time consuming and is not cost effective. Also, augers would have to be withdrawn before installing a monitoring well, which again, may allow the hole to collapse. Furthermore, geologic logging by examining the soils brought to the surface is unreliable, and depth to water may be difficult to determine while drilling.~~

~~There would be very few situations where use of a solid stem auger would be preferable to other drilling methods. The only practical applications of this method would be to drill boreholes for well installation where no lithologic information is desired and the soils are such that the borehole can be expected to remain open after the augers are withdrawn. Alternatively, this technique can be used to find depth to bedrock in an area when no other information is required from drilling.~~

5.2.3 Rotary Drilling

Direct rotary drilling includes air-rotary and fluid-rotary drilling. For air or fluid-rotary drilling, the rotary drill may be advanced to the desired depth by any power-operated drilling machine having sufficient torque

- In some borings temporary casing may not be needed as the drilling fluids may keep the borehole open.
- Drill rigs are readily available in most areas.

Disadvantages to this method include:

- Formation logging is not as accurate as with hollow-stem auger method if split-barrel (split-spoon) samples are not taken (i.e., the depths of materials logged from cuttings delivered to the surface are approximate).
- Drilling fluids reduce permeability of the formation adjacent to the boring to some degree, and require more extensive well development than "dry" techniques (augering, air-rotary).
- No information on depth to water is obtainable while drilling.
- Fluids are needed for drilling, and there is some question about the effects of the drilling fluids on subsequent water samples obtained. For this reason as well, extensive well development may be required.
- In very porous materials (i.e., rubble fill, boulders, coarse gravel) drilling fluids may be continuously lost into the formation. This requires either constant replenishment of the drilling fluid, or the use of casing through this formation.
- Drill rigs are large and heavy, and must be supported with supplied water.
- Groundwater samples can be potentially diluted with drilling fluid.

The procedures for performing direct rotary soil investigations and sampling shall conform with the applicable ASTM standards: D2113-83, D1587-83, and D1586-84.

Soil samples shall be taken as specified by project plan documents, or more frequently, if requested by the project geologist. Any required sampling shall be performed by rotation, pressing, or driving in accordance with the standard or approved method governing use of the particular sampling tool.

When field conditions prevent the advancement of the hole to the desired depth, a new boring may be drilled at the request of the Field Operations Leader. The original boring shall be backfilled using methods and materials appropriate for the given site and a new boring started a short distance away at a location determined by the project geologist.

~~5.2.4 Rotosonic Drilling~~

~~The Rotosonic drilling method employs a high frequency vibrational and low speed rotational motion coupled with down pressure to advance the cutting edge of a drill string. This produces a uniform borehole while providing a continuous, undisturbed core sample of both unconsolidated and most bedrock formations. Rotosonic drilling advances a 4-inch diameter to 12-inch diameter core barrel for sampling and can advance up to a 12-inch diameter outer casing for the construction of standard and telescoped monitoring wells. During drilling, the core barrel is advanced ahead of the outer barrel in increments as determined by the site geologist and depending upon type of material, degree of subsurface contamination and sampling objectives.~~

and ram range to rotate and force the bit to the desired depth. The drilling machine must, however, be equipped with any accessory equipment needed to perform required sampling, or coring. Prior to sampling, any settled drill cuttings in the borehole must be removed.

Air-rotary drilling is a method of drilling where the drill rig simultaneously turns and exerts a downward pressure on the drilling rods and bit while circulating compressed air down the inside of the drill rods, around the bit, and out the annulus of the borehole. Air circulation serves to both cool the bit and remove the cuttings from the borehole. Advantages of this method include:

- The drilling rate is high (even in rock).
- The cost per foot of drilling is relatively low.
- Air-rotary rigs are common in most areas.
- No drilling fluid is required (except when water is injected to keep down dust).
- The borehole diameter is large, to allow room for proper well installation procedures.

Disadvantages to using this method include:

- Formations must be logged from the cuttings that are blown to the surface and thus the depths of materials logged are approximate.
- Air blown into the formation during drilling may "bind" the formation and impede well development and natural groundwater flow.
- In-situ samples cannot be taken, unless the hole is cased.
- Casing must generally be used in unconsolidated materials.
- Air-rotary drill rigs are large and heavy.
- Large amounts of Investigation Derived Waste (IDW) may be generated which may require containerization, sampling, and off-site disposal.

A variation of the typical air-rotary drill bit is a down hole hammer which hammers the drill bit down as it drills. This makes drilling in hard rock faster. Air-rotary drills can also be adapted to use for rock coring although they are generally slower than other types of core drills. A major application of the air-rotary drilling method would be to drill holes in rock for well installation.

Fluid-Rotary drilling operates in a similar manner to air-rotary drilling except that a drilling fluid ("mud") or clean water is used in place of air to cool the drill bit and remove cuttings. There are a variety of fluids that can be used with this drilling method, including bentonite slurry and synthetic slurries. If a drilling fluid other than water/cuttings is used, it must be a natural clay (i.e., bentonite) and a "background" sample of the fluid should be taken for analysis of possible organic or inorganic contaminants.

Advantages to the fluid-rotary drilling method include:

- The ability to drill in many types of formations.
- Relatively quick and inexpensive.
- Split-barrel (split-spoon) or thin-wall (Shelby) tube samples can be obtained without removing drill rods if the appropriate size drill rods and bits (i.e., fish-tail or drag bit) are used.

The outer casing can be advanced at the same time as the inner drill string and core barrel, or advanced down over the inner drill rods and core barrel, or after the core barrel has moved ahead to collect the undisturbed sample and has been pulled out of the borehole. The outer casing can be advanced dry in most cases, or can be advanced with water or air depending upon the formations being drilled, the depth and diameter of the hole, or requirements of the project.

Advantages of this method include:

- Sampling and well installation are faster as compared to other drilling methods.
- Continuous sampling, with larger sample volume as compared to split-spoon sampling.
- The ability to drill through difficult formations such as cobbles or boulders, hard till and bedrock.
- Reduction of IDW by an average of 70 to 80 percent.
- Well installations are quick and controlled by elimination of potential bridging of annular materials during well installation, due to the ability to vibrate the outer casing during removal.

Disadvantages include:

- The cost for Rotosonic drilling as compared to other methods are generally higher. However, the net result can be a significant savings considering reduced IDW and shortened project duration.
- Rotosonic drill rigs are large and need ample room to drill, however, Rotosonic units can be placed on the ground or placed on an ATV.
- There are a limited number of Rotosonic drilling contractors at the present time.

5.2.5 ~~Reverse Circulation Rotary Drilling~~

~~The common reverse circulation rig is a water or mud rotary rig with a large diameter drill pipe which circulates the drilling water down the annulus and up the inside of the drill pipe (reverse flow direction from direct mud rotary). This type of rig is used for the construction of large capacity production water wells and is not suited for small, water quality sampling wells because of the use of drilling muds and the large diameter hole which is created. A few special reverse circulation rotary rigs are made with double wall drill pipe. The drilling water or air is circulated down the annulus between the drill pipes and up inside the inner pipe.~~

~~Advantages of the latter method include:~~

- ~~The formation water is not contaminated by the drilling water.~~
- ~~Formation samples can be obtained, from known depths.~~
- ~~When drilling with air, immediate information is available regarding the water bearing properties of formations penetrated.~~
- ~~Collapsing of the hole in unconsolidated formations is not as great a problem as when drilling with the normal air rotary rig.~~

~~Disadvantages include:~~

- ~~Double-wall, reverse-circulation drill rigs are rare and expensive to operate.~~
- ~~Placing cement grout around the outside of the well casing above a well screen often is difficult, especially when the screen and casing are placed down through the inner drill pipe before the drill pipe is pulled out.~~

5.2.6 Drill-through Casing Driver

The driven-casing method consists of alternately driving casing (fitted with a sharp, hardened casing shoe) into the ground using a hammer lifted and dropped by the drill rig (or an air-hammer) and cleaning out the casing using a rotary chopping bit and air or water to flush out the materials. The casing is driven down in stages (usually 5 feet per stage); a continuous record is kept of the blows per foot in driving the casing (see SOP GH-1.5). The casing is normally advanced by a 300-pound hammer falling freely through a height of 30 inches. Simultaneous washing and driving of the casing is not recommended. If this procedure is used, the elevations within which wash water is used and in which the casing is driven must be clearly recorded.

The driven casing method is used in unconsolidated formations only. When the boring is to be used for later well installation, the driven casing used should be at least 4 inches larger in diameter than the well casing to be installed. Advantages to this method of drilling include:

- Split-barrel (split-spoon) sampling can be conducted while drilling.
- Well installation is easily accomplished.
- Drill rigs used are relatively small and mobile.
- The use of casing minimizes flow into the hole from upper water-bearing layers; therefore, multiple aquifers can be penetrated and sampled for rough field determinations of some water quality parameters.

Some of the disadvantages include:

- This method can only be used in unconsolidated formations.
- The method is slower than other methods (average drilling progress is 30 to 50 feet per day).
- Maximum depth of the borehole varies with the size of the drill rig and casing diameter used, and the nature of the formations drilled.
- The cost per hour or per foot of drilling may be substantially higher than other drilling methods.
- It is difficult and time consuming to pull back the casing if it has been driven very deep (deeper than 50 feet in many formations).

5.2.7 Cable Tool Drilling

A cable tool rig uses a heavy, solid-steel, chisel-type drill bit ("tool") suspended on a steel cable, which when raised and dropped, chisels or pounds a hole through the soils and rock. Drilling progress may be

expedited by the use of "slip-jars" which serve as a cable-activated down hole percussion device to hammer the bit ahead.

When drilling through the unsaturated zone, some water must be added to the hole. The cuttings are suspended in the water and then bailed out periodically. Below the water table, after sufficient ground water enters the borehole to replace the water removed by bailing, no further water needs to be added. When soft caving formations are encountered, it is usually necessary to drive casing as the hole is advanced to prevent collapse of the hole. Often the drilling can be only a few feet below the bottom of the casing. Because the drill bit is lowered through the casing, the hole created by the bit is smaller than the casing. Therefore, the casing (with a sharp, hardened casing shoe on the bottom) must be driven into the hole (see Section 5.2.5 of this guideline).

Advantages of the cable-tool method include the following:

- Information regarding water-bearing zones is readily available during the drilling. Even relative permeabilities and rough water quality data from different zones penetrated can be obtained by skilled operators.
- The cable-tool rig can operate satisfactorily in all formations, but is best suited for caving, boulder, cobble or coarse gravel type formations (e.g., glacial till) or formations with large cavities above the water table (such as limestones).
- When casing is used, the casing seals formation water out of the hole, preventing down hole contamination and allowing sampling of deeper aquifers for field-measurable water quality parameters.
- Split-barrel (split-spoon) or thin-wall (Shelby) tube samples can be collected through the casing.

Disadvantages include:

- Drilling is slow compared with rotary rigs.
- The necessity of driving the casing in unconsolidated formations requires that the casing be pulled back if exposure of selected water-bearing zones is desired. This process complicates the well completion process and often increases costs. There is also a chance that the casing may become stuck in the hole.
- The relatively large diameters required (minimum of 4-inch casing) plus the cost of steel casing result in higher costs compared to rotary drilling methods where casing is not required (e.g., such use of a hollow-stem auger).
- Cable-tool rigs have largely been replaced by rotary rigs. In some parts of the U.S., availability may be difficult.

5.2.8 ~~Jet Drilling (Washing)~~

~~Jet drilling, which should be used only for piezometer or vadose zone sampler installation, consists of pumping water or drilling mud down through a small diameter (1/2 to 2 inch) standard pipe (steel or PVC). The pipe may be fitted with a chisel bit or a special jetting screen. Formation materials dislodged by the bit and jetting action of the water are brought to the surface through the annulus around the pipe. As the pipe is jetted deeper, additional lengths of pipe may be added at the surface.~~

~~Jet percussion is a variation of the jetting method, in which the casing is driven with a drive weight. Normally, this method is used to place 2-inch diameter casing in shallow, unconsolidated sand formations, but this method has also been used to install 3- to 4-inch diameter casings to a depth of 200 feet.~~

~~Jetting is acceptable in very soft formations, usually for shallow sampling, and when introduction of drilling water to the formation is acceptable. Such conditions would occur during rough stratigraphic investigation or installation of piezometers for water level measurement. Advantages of this method include:~~

- ~~Jetting is fast and inexpensive.~~
- ~~Because of the small amount of equipment required, jetting can be accomplished in locations where access by a normal drilling rig would be very difficult. For example, it would be possible to jet down a well point in the center of a lagoon at a fraction of the cost of using a drill rig.~~
- ~~Jetting numerous well points just into a shallow water table is an inexpensive method for determining the water table contours, hence flow direction.~~

~~Disadvantages include the following:~~

- ~~A large amount of foreign water or drilling mud is introduced above and into the formation to be sampled.~~
- ~~Jetting is usually done in very soft formations which are subject to caving. Because of this caving, it is often not possible to place a grout seal above the screen to assure that water in the well is only from the screened interval.~~
- ~~The diameter of the casing is usually limited to 2 inches.~~
- ~~Jetting is only possible in very soft formations that do not contain boulders or coarse gravel, and the depth limitation is shallow (about 30 feet without jet percussion equipment).~~
- ~~Large quantities of water are often needed.~~

5.2.9 Drilling with a Hand Auger

This method is applicable wherever the formation, total depth of sampling, and the site and groundwater conditions are such as to allow hand auger drilling. Hand augering can also be considered at locations where drill rig access is not possible. All hand auger borings will be performed according to ASTM D1452-80.

Samples should be taken continuously unless otherwise specified by the project plan documents. Any required sampling is performed by rotation, pressing, or driving in accordance with the standard or approved method governing use of the particular sampling tool. Typical equipment used for sampling and advancing shallow "hand auger" holes are Iwan samplers (which are rotated) or post hole diggers (which are operated like tongs). These techniques are slow but effective where larger pieces of equipment do not have access, and where very shallow holes are desired (less than 15 feet). Surficial soils must be composed of relatively soft and non-cemented formations to allow penetration by the auger.

5.2.10 Rock Drilling and Coring

When soil borings cannot be continued using augers or rotary methods due to the hardness of the soil or when rock or large boulders are encountered, drilling and sampling can be performed using a diamond bit corer in accordance with ASTM D2113.

Drilling is done by rotating and applying downward pressure to the drill rods and drill bit. The drill bit is a circular, hollow, diamond-studded bit attached to the outer core barrel in a double-tube core barrel. The use of single-tube core barrels is not recommended, as the rotation of the barrel erodes the sample and limits its use for detailed geological evaluation. Water or air is circulated down through the drill rods and annular space between the core barrel tubes to cool the bit and remove the cuttings. The bit cuts a core out of the rock which rises into an inner barrel mounted inside the outer barrel. The inner core barrel and rock core are removed by lowering a wire line with a coupling into the drill rods, latching onto the inner barrel and withdrawing the inner barrel. A less efficient variation of this method utilizes a core barrel that cannot be removed without pulling all of the drill rods. This variation is practical only if less than 50 feet of core is required.

Core borings are made through the casing used for the soil borings. The casing must be driven and sealed into the rock formation to prevent seepage from the overburden into the hole to be cored (see Section 5.3 of this guideline). A double-tube core barrel with a diamond bit and reaming shell or equivalent should be used to recover rock cores of a size specified in the project plans. The most common core barrel diameters are listed in Attachment A.

Soft or decomposed rock should be sampled with a driven split-barrel whenever possible or cored with a Denison or Pitcher sampler.

When coring rock, including shale and claystone, the speed of the drill and the drilling pressure, amount and pressure of water, and length of run can be varied to give the maximum recovery from the rock being drilled. Should any rock formation be so soft or broken that the pieces continually fall into the hole causing unsatisfactory coring, the hole should be reamed and a flush-joint casing installed to a point below the broken formation. The size of the flush-joint casing must permit securing the core size specified. When soft or broken rock is anticipated, the length of core runs should be reduced to less than 5 feet to avoid core loss and minimize core disturbance.

Advantages of core drilling include:

- Undisturbed rock cores can be recovered for examination and/or testing.
- In formations in which the cored hole will remain open without casing, water from the rock fractures may be recovered from the well without the installation of a well screen and gravel pack.
- Formation logging is extremely accurate.
- Drill rigs are relatively small and mobile.

Disadvantages include:

- Water or air is needed for drilling.
- Coring is slower than rotary drilling (and more expensive).
- Depth to water cannot accurately be determined if water is used for drilling.
- The size of the borehole is limited.

This drilling method is useful if accurate determinations of rock lithology are desired or if open wells are to be installed into bedrock. To install larger diameter wells in coreholes, the hole must be reamed out to the proper size after boring, using air or mud rotary drilling methods.

5.2.11 Drilling & Support Vehicles

In addition to the drilling method required to accomplish the objectives of the field program, the type of vehicle carrying the drill rig and/or support equipment and its suitability for the site terrain, will often be an additional deciding factor in planning the drilling program. The types of vehicles available are extensive, and depend upon the particular drilling subcontractor's fleet. Most large drilling subcontractors will have a wide variety of vehicle and drill types suited for most drilling assignments in their particular region, while smaller drilling subcontractors will usually have a fleet of much more limited diversity. The weight, size, and means of locomotion (tires, tracks, etc.) of the drill rig must be selected to be compatible with the site terrain to assure adequate mobility between borehole locations. Such considerations also apply to necessary support vehicles used to transport water and/or drilling materials to the drill rigs at the borehole locations. When the drill rigs or support vehicles do not have adequate mobility to easily traverse the site, provisions must be made for assisting equipment, such as bulldozers, winches, timber planking, etc., to maintain adequate progress during the drilling program.

Some of the typical vehicles which are usually available for drill rigs and support equipment are:

- Totally portable drilling/sampling equipment, where all necessary components (tripods, samplers, hammers, catheads, etc.) may be hand carried to the borehole site. Drilling/sampling methods used with such equipment include:
  - Hand augers and lightweight motorized augers.
  - Retractable plug samplers--driven by hand (hammer).
  - Motorized cathead - a lightweight aluminum tripod with a small gas-engine cathead mounted on one leg, used to install small-diameter cased borings. This rig is sometimes called a "monkey on a stick."
- Skid-mounted drilling equipment containing a rotary drill or engine-driven cathead (to lift hammers and drill string), a pump, and a dismounted tripod. The skid is pushed, dragged, or winched (using the cathead drum) between boring locations.
- Small truck-mounted drilling equipment using a Jeep, stake body or other light truck (4 to 6 wheels), upon which are mounted the drill and/or a cathead, a pump, and a tripod or small drilling derrick. On some rigs, the drill and/or a cathead are driven by a power take-off from the truck, instead of by a separate engine.
- Track-mounted drilling equipment is similar to truck-mounted rigs, except that the vehicle used has wide bulldozer tracks for traversing soft ground. Sometimes a continuous-track "all terrain vehicle" is also modified for this purpose. Some types of tracked drill rigs are called "bombardier" or "weasel" rigs.
- Heavy truck-mounted drilling equipment is mounted on tandem or dual tandem trucks to transport the drill, derrick, winches, and pumps or compressors. The drill may be provided with a separate engine or may use a power take-off from the truck engine. Large augers, hydraulic rotary and reverse circulation rotary drilling equipment are usually mounted on such heavy duty trucks. For soft-ground sites, the drilling equipment is sometimes mounted on vehicles having low pressure, very wide diameter tires and capable of floating; these vehicles are called "swamp buggy" rigs.

- Marine drilling equipment is mounted on various floating equipment for drilling borings in lakes, estuaries and other bodies of water. The floating equipment varies, and is often manufactured or customized by the drilling subcontractor to suit specific drilling requirements. Typically, the range of flotation vehicles include:

  - Barrel float rigs – a drill rig mounted on a timber platform buoyed by empty 55-gallon drums or similar flotation units.

  - Barge-mounted drill rigs.

  - Jack-up platforms – drilling equipment mounted on a floating platform having retractable legs to support the unit on the sea or lake bed when the platform is jacked up out of the water.

  - Drill ships – for deep ocean drilling.

In addition to the mobility for the drilling equipment, similar consideration must be given for equipment to support the drilling operations. Such vehicles or floating equipment are needed to transport drill water, drilling supplies and equipment, samples, drilling personnel, etc. to and/or from various boring locations.

5.2.12 Equipment Sizes

In planning subsurface exploration programs, care must be taken in specifying the various drilling components, so that they will fit properly in the boring or well.

For drilling open boreholes using rotary drilling equipment, tri-cone drill bits are employed with air, water or drilling mud to remove cuttings and cool the bit. Tri-cone bits are slightly smaller than the holes they drill (i.e., 5-7/8-inch or 7-7/8-inch bits will nominally drill 6-inch and 8-inch holes, respectively).

For obtaining split-barrel samples of a formation, samplers are commonly manufactured in sizes ranging from 2 inches to 3-1/2 inches in outside diameter. However, the most commonly used size is the 2-inch O.D., 1-3/8-inch I.D. split-barrel sampler. When this sampler is used and driven by a 140-pound (± 2-pound) hammer dropping 30 inches (± 1 inch), the procedure is called a Standard Penetration Test, and the blows per foot required to advance the sampler into the formation can be correlated to the formation's density or strength.

In planning the drilling of boreholes using hollow-stem augers or casing, in which thin-wall tube samples or diamond core drilling will be performed, refer to the various sizes and clearances provided in Attachment A of this guideline. Sizes selected must be stated in the project plan documents.

5.2.13 Estimated Drilling Progress

To estimate the anticipated rates of drilling progress for a site, the following must be considered:

- The speed of the drilling method employed.
- Applicable site conditions (e.g., terrain, mobility between borings, difficult drilling conditions in bouldery soils, rubble fill or broken rock, etc.).
- Project-imposed restrictions (e.g., drilling while wearing personal protective equipment, decontamination of drilling equipment, etc.).

Based on recent experience in drilling average soil conditions (no boulders) and taking samples at 5-foot intervals, for moderate depth (30 feet to 50 feet) boreholes (not including installation or development of wells), the following daily rates of total drilling progress may be anticipated for the following drilling methods:

Drilling Method	Average Daily Progress (linear feet)
Hollow-stem augers	75'
Solid-stem augers	50'
Mud-Rotary Drilling	100' (cuttings samples)
Rotosonic Drilling	100'-160' (continuous core)
Reverse-Circulation Rotary	100' (cuttings samples)
Skid-Rig with driven casing	30'
Rotary with driven casing	50'
Cable Tool	30'
Hand Auger	Varies
Continuous Rock Coring	50'

5.3 Prevention of Cross-Contamination

A telescoping or multiple casing technique minimizes the potential for the migration of contaminated groundwater to lower strata below a confining layer. The telescoping technique consists of drilling to a confining layer utilizing a spun casing method with a diamond cutting or augering shoe (a method similar to the rock coring method described in Section 5.2.10, except that larger casing is used) or by using a driven-casing method (see Section 5.2.6 of this guideline) and installing a specified diameter steel well casing. The operation consists of three separate steps. Initially, a drilling casing (usually of 8-inch diameter) is installed followed by installation of the well casing (6-inch-diameter is common for 2-inch wells). This well casing is driven into the confining layer to ensure a tight seal at the bottom of the hole. The well casing is sealed at the bottom with a bentonite-cement slurry. The remaining depth of the boring is drilled utilizing a narrower diameter spun or driven casing technique within the outer well casing. A smaller diameter well casing with an appropriate length of slotted screen on the lower end, is installed to the surface.

Clean sand is placed in the annulus around and to a point of about 2 feet above the screen prior to withdrawal of the drilling casing. The annular space above the screen and to a point 2 feet above the bottom of the outer well casing is sealed with a tremied cement-bentonite slurry which is pressure-grouted or displacement-grouted into the hole. The remaining casing annulus is backfilled with clean material and grouted at the surface, or it is grouted all the way to the surface.

5.4 Cleanout of Casing Prior to Sampling

The boring hole must be completely cleaned of disturbed soil, segregated coarse material and clay adhering to the inside walls of the casing. The cleaning must extend to the bottom edge of the casing and, if possible, a short distance further (1 or 2 inches) to bypass disturbed soil resulting from the advancement of the casing. Loss of wash water during cleaning should be recorded.

For disturbed samples both above and below the water table and where introduction of relatively large volumes of wash water is permissible, the cleaning operation is usually performed by washing the material out of the casing with water; however, the cleaning should never be accomplished with a strong, downward-directed jet which will disturb the underlying soil. When clean out has reached the bottom of the casing or slightly below (as specified above), the string of tools should be lifted one foot off the bottom with the water still flowing, until the wash water coming out of the casing is clear of granular soil particles. In formations where the cuttings contain gravel and other larger particles, it is often useful to repeatedly raise and lower the drill rods and wash bit while washing out the hole, to surge these large particles upward out of the hole. As a time saver, the drilling contractor may be permitted to use a split-barrel (split-spoon) sampler with the ball check valve removed as the clean-out tool, provided the material below the spoon is not disturbed and the shoe of the spoon is not damaged. However, because the ball check valve has been removed, in some formations it may be necessary to install a flap valve or spring sample retainer in the split-spoon bit, to prevent the sample from falling out as the sampler is withdrawn from the hole. The use of jet-type chopping bits is discouraged except where large boulders and cobbles or hard-cemented soils are encountered. If water markedly softens the soils above the water table, clean out should be performed dry with an auger.

For undisturbed samples below the water table, or where wash water must be minimized, clean out is usually accomplished with an appropriate diameter clean out auger. This auger has cutting blades at the bottom to carry loose material up into the auger, and up-turned water jets just above the cutting blades to carry the removed soil to the surface. In this manner, there is a minimum of disturbance at the top of the material to be sampled. If any gravel material washes down into the casing and cannot be removed by the clean out auger, a split-barrel sample can be taken to remove it; bailers and sandpumps should not be used. For undisturbed samples above the groundwater table, all operations must be performed in a dry manner.

If all of the cuttings created by drilling through the overlying formations are not cleaned from the borehole prior to sampling, some of the problems which may be encountered during sampling include:

- When sampling is attempted through the cuttings remaining in the borehole, all or part of the sampler may become filled with the cuttings. This limits the amount of sample from the underlying formation which can enter and be retained in the sampler, and also raises questions as to the validity of the sample.
- If the cuttings remaining in the borehole contain coarse gravel and/or other large particles, these may block the bit of the sampler and prevent any materials from the underlying formation from entering the sampler when the sampler is advanced.
- In cased borings, should sampling be attempted through cuttings which remain in the lower portion of the casing, these cuttings could cause the sampler to become bound into the casing, such that it becomes very difficult to either advance or retract the sampler.
- When sampler blow counts are used to estimate the density or strength of the formation being sampled, the presence of cuttings in the borehole will usually give erroneously high sample blow counts.

To confirm that all cuttings have been removed from the borehole prior to attempting sampling, it is important that the site geologist measure the "stickup" of the drill string. This is accomplished by measuring the assembled length of all drill rods and bits or samplers (the drill string) as they are lowered to the bottom of the hole, below some convenient reference point of the drill string, then measuring the height of this reference point above the ground surface. The difference of these measurements is the

depth of the drill string (lower end of the bit or sampler) below the ground surface, which must then be compared with the depth of sampling required (installed depth of casing or depth of borehole drilled). If the length of drill string below grade is more than the drilled or casing depth, the borehole has been cleaned too deeply, and this deeper depth of sampling must be recorded on the log. If the length of drill string below grade is less than the drilled or casing depth, the difference represents the thickness of cuttings which remain in the borehole. In most cases, an inch or two of cuttings may be left in the borehole with little or no problem. However, if more than a few inches of cuttings are encountered, the borehole must be recleaned prior to attempting sampling.

5.5 Materials of Construction

The effects of monitoring well construction materials on specific chemical analytical parameters are described and/or referenced in SOP GH-2.8. However, there are several materials used during drilling, particularly drilling fluids and lubricants, which must be used with care to avoid compromising the representativeness of soil and ground water samples.

The use of synthetic or organic polymer slurries is not permitted at any location where soil samples for chemical analysis are to be collected. These slurry materials could be used for installation of long-term monitoring wells, but the early time data in time series collection of ground water data may then be suspect. If synthetic or organic polymer muds are proposed for use at a given site, a complete written justification including methods and procedures for their use must be provided by the site geologist and approved by the Project Manager. The specific slurry composition and the concentration of suspected contaminants for each site must be known.

For many drilling operations, potable water is an adequate lubricant for drill stem and drilling tool connections. However, there are instances, such as drilling in tight clayey formations or in loose gravels, when threaded couplings must be lubricated to avoid binding. In these instances, to be determined in the field by the judgment of the site geologist and noted in the site logbook, and only after approval by the Project Manager, a vegetable oil or silicone-based lubricant should be used. Petroleum based greases, etc. will not be permitted. Samples of lubricants used must be provided and analyzed for chemical parameters appropriate to the given site.

5.6 Subsurface Soil Samples

Subsurface soil samples are used to characterize subsurface stratigraphy. This characterization can indicate the potential for migration of chemical contaminants in the subsurface. In addition, definition of the actual migration of contaminants can be obtained through chemical analysis of the soil samples. Where the remedial activities may include in-situ treatment or excavation and removal of the contaminated soil, the depth and areal extent of contamination must be known as accurately as possible.

Engineering and physical properties of soil may also be of interest should site construction activities be planned. Soil types, grain size distribution, shear strength, compressibility, permeability, plasticity, unit weight, and moisture content are some of the physical characteristics that may be determined for soil samples.

Penetration tests are also described in this procedure. The tests can be used to estimate various physical and engineering parameters such as relative density, unconfined compressive strength, and consolidation characteristics of soils.

Surface protocols for various soil sampling techniques are discussed in SOP SA-1.3. Continuous-core soil sampling and rock coring are discussed below. The procedures described here are representative of

a larger number of possible drilling and sampling techniques. The choice of techniques is based on a large number of variables such as cost, local geology, etc. The final choice of methods must be made with the assistance of drilling subcontractors familiar with the local geologic conditions. Alternative techniques must be based upon the underlying principles of quality assurance implicit in the following procedures.

The CME continuous sample tube system provides a method of sampling soil continuously during hollow-stem augering. The 5-foot sample barrel fits within the lead auger of a hollow-auger column. The sampling system can be used with a wide range of I.D. hollow-stem augers (from 3-1/4-inch to 8-1/4-inch I.D.). This method has been used to sample many different materials such as glacial drift, hard clays and shales, mine tailings, etc. This method is particularly used when SPT samples are not required and a large volume of material is needed. Also, this method is useful when a visual description of the subsurface lithology is required. Rotosonic drilling methods also provide a continuous soil sample.

5.7 Rock Sampling (Coring) (ASTM D2113-83)

Rock coring enables a detailed assessment of borehole conditions to be made, showing precisely all lithologic changes and characteristics. Because coring is an expensive drilling method, it is commonly used for shallow studies of 500 feet or less, or for specific intervals in the drill hole that require detailed logging and/or analyzing. Rock coring can, however, proceed for thousands of feet continuously, depending on the size of the drill rig, and yields better quality data than air-rotary drilling, although at a substantially reduced drilling rate. Rate of drilling varies widely, depending on the characteristics of lithologies encountered, drilling methods, depth of drilling, and condition of drilling equipment. Average output in a 10-hour day ranges from 40 to over 200 feet. Down hole geophysical logging or television camera monitoring is sometimes used to complement the data generated by coring.

Borehole diameter can be drilled to various sizes, depending on the information needed. Standard sizes of core barrels (showing core diameter) and casing are shown in Figure 1.

Core drilling is used when formations are too hard to be sampled by soil sampling methods and a continuous solid sample is desired. Usually, soil samples are used for overburden, and coring begins in sound bedrock. Casing is set into bedrock before coring begins to prevent loose material from entering the borehole, to prevent loss of drilling fluid, and to prevent cross-contamination of aquifers.

Drilling through bedrock is initiated by using a diamond-tipped core bit threaded to a drill rod (outer core barrel) with a rate of drilling determined by the downward pressure, rotation speed of drill rods, drilling fluid pressure in the borehole, and the characteristics of the rock (mineralogy, cementation, weathering).

FIGURE 1

STANDARD SIZES OF CORE BARRELS AND CASING

Coring Bit Size	Nominal*		Set Size*	
	O.D.	I.D.	O.D.	I.D.
RWT	1 5/32	3/4	1.160	0.735
EWT	1 1/2	29/32	1.470	0.905
EX, EXL, EWG, EWM	1 1/2	13/16	1.470	0.845
AWT	1 7/8	1 9/32	1.875	1.281
AX, AXL, AWG, AWM	1 7/8	1 3/16	1.875	1.185
BWT	2 3/8	1 3/4	2.345	1.750
BX, BXL, BWG, BWM	2 3/8	1 5/8	2.345	1.655
NWT	3	2 5/16	2.965	2.313
NX, NXL, NWG, NWM	3	2 1/8	2.965	2.155
HWT	3 29/32	3 3/16	3.889	3.187
HWG	3 29/32	3	3.889	3.000
2 3/4 x 3 7/8	3 7/8	2 3/4	3.840	2.690
4 x 5 1/2	5 1/2	4	5.435	3.970
6 x 7 3/4	7 3/4	6	7.655	5.970
AX Wire line ___|___/	1 7/8	1	1.875	1.000
BX Wire line ___|___/	2 3/8	1 7/16	2.345	1.437
NX Wire line ___|___/	3	1 15/16	2.965	1.937

* All dimensions are in inches; to convert to millimeters, multiply by 25.4.

___|___/ Wire line dimensions and designations may vary according to manufacturer.

FIGURE 1
STANDARD SIZES OF CORE BARRELS AND CASING
PAGE TWO

Size Designations		Casing O.D., Inches	Casing Coupling		Casing bit O.D., Inches	Core barrel bit O.D., Inches*	Drill rod O.D., Inches	Approximate Core Diameter	
Casing; Casing coupling; Casing bits; Core barrel bits	Rod; rod couplings		O.D., Inches	I.D., Inches				Normal, Inches	Thinwall, Inches
RX	RW	1.437	1.437	1.188	1.485	1.160	1.094	—	0.735
EX	E	1.812	1.812	1.500	1.875	1.470	1.313	0.845	0.905
AX	A	2.250	2.250	1.906	2.345	1.875	1.625	1.185	1.281
BX	B	2.875	2.875	2.375	2.965	2.345	1.906	1.655	1.750
NX	N	3.500	3.500	3.000	3.615	2.965	2.375	2.155	2.313
HX	HW	4.500	4.500	3.938	4.625	3.890	3.500	3.000	3.187
RW	RW	1.437	Flush Joint	No Coupling	1.485	1.160	1.094	—	0.735
EW	EW	1.812			1.875	1.470	1.375	0.845	0.905
AW	AW	2.250			2.345	1.875	1.750	1.185	1.281
BW	BW	2.875			2.965	2.345	2.125	1.655	1.750
NW	NW	3.500			3.615	2.965	2.625	2.155	2.313
HW	HW	4.500			4.625	3.890	3.500	3.000	3.187
PW	—	5.500			5.650	—	—	—	—
SW	—	6.625			6.790	—	—	—	—
UW	—	7.625			7.800	—	—	—	—
ZW	—	8.625			8.810	—	—	—	—
—	AX__|__\	—	—	—	—	1.875	1.750	1.000	—
—	BX__|__\	—	—	—	—	2.345	2.250	1.437	—
—	NX__|__\	—	—	—	—	2.965	2.813	1.937	—

* All dimensions are in inches; to convert to millimeters, multiply by 25.4.

__\|__/ Wire line dimensions and designations may vary according to manufacturer.

NOMINAL DIMENSIONS FOR DRILL CASINGS AND ACCESSORIES. (DIAMOND CORE DRILL MANUFACTURERS ASSOCIATION). 288-D-2889

5.7.1 Diamond Core Drilling

A penetration of typically less than 6 inches per 50 blows using a 140-lb. hammer dropping 30 inches with a 2-inch split-barrel sampler shall be considered an indication that soil sampling methods may not be applicable and that coring may be necessary to obtain samples.

When formations are encountered that are too hard to be sampled by soil sampling methods, the following diamond core drilling procedure may be used:

- Firmly seat a casing into the bedrock or the hard material to prevent loose materials from entering the hole and to prevent the loss of drilling fluid return. Level the surface of the rock or hard material when necessary by the use of a fishtail or other bits. If the drill hole can be retained open without the casing and if cross-contamination of aquifers in the unconsolidated materials is unlikely, leveling may be omitted.

- Begin the core drilling using a double-tube swivel-core barrel of the desired size. After drilling no more than 10 feet (3 m), remove the core barrel from the hole and take out the core. If the core blocks the flow of the drilling fluid during drilling, remove the core barrel immediately. In soft materials, a large starting size may be specified for the coring tools; where local experience indicates satisfactory core recovery or where hard, sound materials are anticipated, a smaller size or the single-tube type may be specified and longer runs may be drilled. NX/NW size coring equipment is the most commonly used size.

- When soft materials are encountered that produce less than 50 percent recovery, stop the core drilling. If soil samples are desired, secure such samples in accordance with the procedures described in ASTM Method D 1586 (Split-barrel Sampling) or in Method D 1587 (Thin-Walled Tube Sampling); sample soils per SOP SA-1.3. Resume diamond core drilling when refusal materials are again encountered.

- Since rock structures and the occurrence of seams, fissures, cavities, and broken areas are among the most important items to be detected and described, take special care to obtain and record these features. If such broken zones or cavities prevent further advance of the boring, one of the following three steps shall be taken: (1) cement the hole; (2) ream and case; or (3) case and advance with the next smaller size core barrel, as conditions warrant.

- In soft, seamy, or otherwise unsound rock, where core recovery may be difficult, M-design core barrels may be used. In hard, sound rock where a high percentage of core recovery is anticipated, the single-tube core barrel may be employed.

5.7.2 Rock Sample Preparation and Documentation

Once the rock coring has been completed and the core recovered, the rock core shall be carefully removed from the barrel, placed in a core tray (previously labeled "top" and "bottom" to avoid confusion), classified, and measured for percentage of recovery as well as the rock quality designation (RQD). Each core shall be described, classified, and logged using a uniform system as presented in SOP GH-1.5. If moisture content will be determined or if it is desirable to prevent drying (e.g., to prevent shrinkage of clay formations) or oxidation of the core, the core shall be wrapped in plastic sleeves immediately after logging. Each plastic sleeve shall be labeled with indelible ink. The boring number, run number, and the footage represented in each sleeve shall be included, as well as designating the top and bottom of the core run.

After sampling, rock cores shall be placed in the sequence of recovery in well-constructed wooden boxes provided by the drilling contractor. Rock cores from two different borings shall not be placed in the same core box unless accepted by the Project Geologist. The core boxes shall be constructed to accommodate at least 20 linear feet of core in rows of approximately 5 feet each and shall be constructed with hinged tops secured with screws, and a latch (usually a hook and eye) to keep the top securely fastened down. Wood partitions shall be placed at the end of each core run and between rows.

The depth from the surface of the boring to the top and bottom of the drill run and run number shall be marked on the wooden partitions with indelible ink. A wooden partition (wooden block) shall be placed at the end of each run with the depth of the bottom of the run written on the block. These blocks will serve to separate successive core runs and indicate depth intervals for each run. The order of placing cores shall be the same in all core boxes. Rock core shall be placed in the box so that, when the box is open, with the inside of the lid facing the observer, the top of the cored interval contained within the box is in the upper left corner of the box, and the bottom of the cored interval is in the lower right corner of the box. The top and bottom of each core obtained and its true depth shall be clearly and permanently marked on each box. The width of each row must be compatible with the core diameter to prevent lateral movement of the core in the box. Similarly, an empty space in a row shall be filled with an appropriate filler material or spacers to prevent longitudinal movement of the core in the box.

The inside and outside of the core-box lid shall be marked by indelible ink to show all pertinent data on the box's contents. At a minimum, the following information shall be included:

- Project name.
- Project number.
- Boring number.
- Run numbers.
- Footage (depths).
- Recovery.
- RQD (%).
- Box number and total number of boxes for that boring (Example: Box 5 of 7).

For easy retrieval when core boxes are stacked, the sides and ends of the box shall also be labeled and include project number, boring number, top and bottom depths of core and box number.

Prior to final closing of the core box, a photograph of the recovered core and the labeling on the inside cover shall be taken. If moisture content is not critical, the core shall be wetted and wiped clean for the photograph. (This will help to show true colors and bedding features in the cores).

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ATTACHMENT A

DRILLING EQUIPMENT SIZES

Drilling Component	Designation or Hole Size (Inches)	O.D. (Inches)	I.D. (Inches)	Coupling I.D. (Inches)
Hollow-stem augers (Ref. 7)	6 1/4	5	2 1/4	
	6 3/4	5 3/4	2 3/4	—
	7 1/4	6 1/4	3 1/4	—
	13 1/4	12	6	—
Thin Wall Tube Samplers (Ref. 7)	—	2	1 7/8	—
	—	2 1/2	2 3/8	—
	—	3	2 7/8	—
	—	3 1/2	3 3/8	—
	—	4 1/2	4 3/8	—
	—	5	4 3/4	—
Drill Rods (Ref. 7)	RW	1 3/32	23/32	13/32
	EW	1 3/8	15/16	7/16
	AW	1 3/4	1 1/4	5/8
	BW	2 1/8	1 3/4	3/4
	NW	2 5/8	2 1/4	1 3/8
	HW	3 1/2	3 1/16	2 3/8
	E	1 5/16	7/8	7/16
	A	1 5/8	1 1/8	9/16
	B	1 7/8	1 1/4	5/8
	N	2 3/8	2	1
				Wall Thickness (Inches)
Driven External Coupled Extra Strong Steel* Casing (Ref. 8)	2 1/2	2.875	2.323	0.276
	3	3.5	2.9	0.300
	3 1/2	4.0	3.364	0.318
	4	4.5	3.826	0.337
	5	5.63	4.813	0.375
	6	6.625	5.761	0.432
	8	8.625	7.625	0.500
	10	10.750	9.750	0.500
	12	12.750	11.750	0.500

* Add twice the casing wall thickness to casing O.D. to obtain the approximate O.D. of the external pipe couplings.

ATTACHMENT A
DRILLING EQUIPMENT SIZES
PAGE TWO

Drilling Component	Designation or Hole Size (Inches)	O.D. (Inches)	I.D. (Inches)	Coupling I.D. (Inches)
Flush Coupled Casing (Ref. 7)	RX	1 7/16	1 3/16	1 3/16
	EX	1 13/16	1 5/8	1 1/2
	AX	2 1/4	2	1 29/32
	BX	2 7/8	2 9/16	2 3/8
	NX	3 1/2	3 3/16	3
	HX	4 1/2	4 1/8	3 15/16
Flush Joint Casing (Ref. 7)	RW	1 7/16	1 3/16	
	EW	1 13/16	1 1/2	
	AW	2 1/4	1 29/32	
	BW	2 7/8	2 3/8	
	NW	3 1/2	3	
	HW	4 1/2	4	
	PW	5 1/2	5	
	SW	6 5/8	6	
	UW	7 5/8	7	
	ZW	8 5/8	8	
Diamond Core Barrels (Ref. 7)	EWM	1 1/2	7/8**	
	AWM	1 7/8	1 1/8**	
	BWM	2 3/8	1 5/8**	
	NWM	3	2 1/8	
	HWG	3 7/8	3	
	2 3/4 x 3 7/8	3 7/8	2 11/16	
	4 x 5 1/2	5 1/2	3 15/16	
	6 x 7 3/4	7 3/4	5 15/16	
	AQ (wireline)	1 57/64	1 1/16**	
	BQ (wireline)	2 23/64	1 7/16**	
	NQ (wireline)	2 63/64	1 7/8	
	HQ (wireline)	3 25/32	2 1/2	

** Because of the fragile nature of the core and the difficulty to identify rock details, use of small-diameter core (1 3/8") is not recommended.

TETRA TECH NUS, INC.	STANDARD OPERATING PROCEDURES	Number GH-1.5	Page 1 of 20
		Effective Date 06/99	Revision 1
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject BOREHOLE AND SAMPLE LOGGING		Approved D. Senovich	

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1.0 PURPOSE

The purpose of this document is to establish standard procedures and technical guidance on borehole and sample logging.

2.0 SCOPE

These procedures provide descriptions of the standard techniques for borehole and sample logging. These techniques shall be used for each boring logged to provide consistent descriptions of subsurface lithology. While experience is the only method to develop confidence and accuracy in the description of soil and rock, the field geologist/engineer can do a good job of classification by careful, thoughtful observation and by being consistent throughout the classification procedure.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES

Site Geologist. Responsible for supervising all boring activities and assuring that each borehole is completely logged. If more than one rig is being used on site, the Site Geologist must make sure that each field geologist is properly trained in logging procedures. A brief review or training session may be necessary prior to the start up of the field program and/or upon completion of the first boring.

5.0 PROCEDURES

The classification of soil and rocks is one of the most important jobs of the field geologist/engineer. To maintain a consistent flow of information, it is imperative that the field geologist/engineer understand and accurately use the field classification system described in this SOP. This identification is based on visual examination and manual tests.

5.1 Materials Needed

When logging soil and rock samples, the geologist or engineer may be equipped with the following:

- Rock hammer
- Knife
- Camera
- Dilute hydrochloric acid (HCl)
- Ruler (marked in tenths and hundredths of feet)
- Hand Lens

5.2 Classification of Soils

All data shall be written directly on the boring log (Figure 1) or in a field notebook if more space is needed. Details on filling out the boring log are discussed in Section 5.5.

FIGURE 1

BORING LOG (EXAMPLE)

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING NUMBER: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	Color	Material Classification	USCS*	Remarks	PID/FID Reading (ppm): Sample	Sampler BZ	Borehole**	Drillers BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 5 foot intervals @ borehole. Increase reading frequency if elevated response read.

Drilling Area Background (ppm): ☐

Remarks: _____

Converted to Well: Yes _____ No _____ Well I.D. #: _____

FIGURE 1 (CONTINUED)

5.2.1 USCS Classification

Soils are to be classified according to the Unified Soil Classification System (USCS). This method of classification is detailed in Figure 1 (Continued).
This method of classification identifies soil types on the basis of grain size and cohesiveness.

Fine-grained soils, or fines, are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no size range; it is recognized by its composition. The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

Coarse-grained soils shall be divided into rock fragments, sand, or gravel. The terms sand and gravel not only refer to the size of the soil particles but also to their depositional history. To insure accuracy in description, the term rock fragments shall be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges typically observed indicate little or no transport from their source area, and therefore the term provides additional information in reconstructing the depositional environment of the soils encountered. When the term "rock fragments" is used it shall be followed by a size designation such as "(1/4 inchΦ-1/2 inchΦ)" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

5.2.2 Color

Soil colors shall be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Since color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

Colors must be described while the sample is still moist. Soil samples shall be broken or split vertically to describe colors. Samplers tend to smear the sample surface creating color variations between the sample interior and exterior.

The term "mottled" shall be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

Soil Color Charts shall not be used unless specified by the project manager.

5.2.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the geologist is to first identify the soil type. Granular soils contain predominantly sands and gravels. They are noncohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

The density of noncohesive, granular soils is classified according to standard penetration resistances obtained from split-barrel sampling performed according to the methods detailed in Standard Operating Procedures GH-1.3 and SA-1.3. Those designations are:

Designation	Standard Penetration Resistance (Blows per Foot)
Very loose	0 to 4
Loose	5 to 10
Medium dense	11 to 30
Dense	31 to 50
Very dense	Over 50

Standard penetration resistance is the number of blows required to drive a split-barrel sampler with a 2-inch outside diameter 12 inches into the material using a 140-pound hammer falling freely through 30 inches. The sampler is driven through an 18-inch sample interval, and the number of blows is recorded for each 6-inch increment. The density designation of granular soils is obtained by adding the number of blows required to penetrate the last 12 inches of each sample interval. It is important to note that if gravel or rock fragments are broken by the sampler or if rock fragments are lodged in the tip, the resulting blow count will be erroneously high, reflecting a higher density than actually exists. This shall be noted on the log and referenced to the sample number. Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in Figure 2.

Cohesive soils are given the USCS classifications ML, MH, CL, CH, OL, or OH (see Figure 1).

The consistency of cohesive soils is determined either by blow counts, a pocket penetrometer (values listed in the table as Unconfined Compressive Strength), or by hand by determining the resistance to penetration by the thumb. The pocket penetrometer and thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample in the split-barrel sampler. The sample shall be broken in half and the thumb or penetrometer pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. Consistency shall not be determined solely by blow counts. One of the other methods shall be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in Figure 2.

5.2.4 Weight Percentages

In nature, soils are comprised of particles of varying size and shape, and are combinations of the various grain types. The following terms are useful in the description of soil:

Terms of Identifying Proportion of the Component	Defining Range of Percentages by Weight
Trace	0 - 10 percent
Some	11 - 30 percent
Adjective form of the soil type (e.g., "sandy")	31 - 50 percent

FIGURE 2

CONSISTENCY FOR COHESIVE SOILS

Consistency	Standard Penetration Resistance (Blows per Foot)	Unconfined Compressive Strength (Tons/Sq. Foot by pocket penetration)	Field Identification
Very soft	0 to 2	Less than 0.25	Easily penetrated several inches by fist
Soft	2 to 4	0.25 to 0.50	Easily penetrated several inches by thumb
Medium stiff	4 to 8	0.50 to 1.0	Can be penetrated several inches by thumb with moderate effort
Stiff	8 to 15	1.0 to 2.0	Readily indented by thumb but penetrated only with great effort
Very stiff	15 to 30	2.0 to 4.0	Readily indented by thumbnail
Hard	Over 30	More than 4.0	Indented with difficulty by thumbnail

Examples:

- Silty fine sand: 50 to 69 percent fine sand, 31 to 50 percent silt.
- Medium to coarse sand, some silt: 70 to 80 percent medium to coarse sand, 11 to 30 percent silt.
- Fine sandy silt, trace clay: 50 to 68 percent silt, 31 to 49 percent fine sand, 1 to 10 percent clay.
- Clayey silt, some coarse sand: 70 to 89 percent clayey silt, 11 to 30 percent coarse sand.

5.2.5 Moisture

Moisture content is estimated in the field according to four categories: dry, moist, wet, and saturated. In dry soil, there appears to be little or no water. Saturated samples obviously have all the water they can hold. Moist and wet classifications are somewhat subjective and often are determined by the individual's judgment. A suggested parameter for this would be calling a soil wet if rolling it in the hand or on a porous surface liberates water, i.e., dirties or muddies the surface. Whatever method is adopted for describing moisture, it is important that the method used by an individual remains consistent throughout an entire drilling job.

Laboratory tests for water content shall be performed if the natural water content is important.

5.2.6 Stratification

Stratification can only be determined after the sample barrel is opened. The stratification or bedding thickness for soil and rock is depending on grain size and composition. The classification to be used for stratification description is shown in Figure 3.

5.2.7 Texture/Fabric/Bedding

The texture/fabric/bedding of the soil shall be described. Texture is described as the relative angularity of the particles: rounded, subrounded, subangular, and angular. Fabric shall be noted as to whether the particles are flat or bulky and whether there is a particular relation between particles (i.e., all the flat particles are parallel or there is some cementation). The bedding or structure shall also be noted (e.g., stratified, lensed, nonstratified, heterogeneous varved).

5.2.8 Summary of Soil Classification

In summary, soils shall be classified in a similar manner by each geologist/engineer at a project site. The hierarchy of classification is as follows:

- Density and/or consistency
- Color
- Plasticity (Optional)
- Soil types
- Moisture content
- Stratification
- Texture, fabric, bedding
- Other distinguishing features

FIGURE 3

BEDDING THICKNESS CLASSIFICATION

Thickness (metric)	Thickness (Approximate English Equivalent)	Classification
> 1.0 meter	> 3.3'	Massive
30 cm - 1 meter	1.0' - 3.3'	Thick Bedded
10 cm - 30 cm	4" - 1.0'	Medium Bedded
3 cm - 10 cm	1" - 4"	Thin Bedded
1 cm - 3 cm	2/5" - 1"	Very Thin Bedded
3 mm - 1 cm	1/8" - 2/5"	Laminated
1 mm - 3 mm	1/32" - 1/8"	Thinly Laminated
< 1 mm	<1/32"	Micro Laminated

(Weir, 1973 and Ingram, 1954)

5.3 Classification of Rocks

Rocks are grouped into three main divisions: sedimentary, igneous and metamorphic. Sedimentary rocks are by far the predominant type exposed at the earth's surface. The following basic names are applied to the types of rocks found in sedimentary sequences:

- Sandstone - Made up predominantly of granular materials ranging between 1/16 to 2 mm in diameter.
- Siltstone - Made up of granular materials less than 1/16 to 1/256 mm in diameter. Fractures irregularly. Medium thick to thick bedded.
- Claystone - Very fine-grained rock made up of clay and silt-size materials. Fractures irregularly. Very smooth to touch. Generally has irregularly spaced pitting on surface of drilled cores.
- Shale - A fissile very fine-grained rock. Fractures along bedding planes.
- Limestone - Rock made up predominantly of calcite ($CaCO_3$). Effervesces strongly upon the application of dilute hydrochloric acid.
- Coal - Rock consisting mainly of organic remains.
- Others - Numerous other sedimentary rock types are present in lesser amounts in the stratigraphic record. The local abundance of any of these rock types is dependent upon the depositional history of the area. Conglomerate, halite, gypsum, dolomite, anhydrite, lignite, etc. are some of the rock types found in lesser amounts.

In classifying a sedimentary rock the following hierarchy shall be noted:

- Rock type
- Color
- Bedding thickness
- Hardness
- Fracturing
- Weathering
- Other characteristics

5.3.1 Rock Type

As described above, there are numerous types of sedimentary rocks. In most cases, a rock will be a combination of several grain types, therefore, a modifier such as a sandy siltstone, or a silty sandstone can be used. The modifier indicates that a significant portion of the rock type is composed of the modifier. Other modifiers can include carbonaceous, calcareous, siliceous, etc.

Grain size is the basis for the classification of clastic sedimentary rocks. Figure 4 is the Udden-Wentworth classification that will be assigned to sedimentary rocks. The individual boundaries are slightly different than the USCS subdivision for soil classification. For field determination of grain sizes, a scale can be used for the coarse grained rocks. For example, the division between siltstone and claystone may not be measurable in the field. The boundary shall be determined by use of a hand lens. If the grains cannot be seen with the naked eye but are distinguishable with a hand lens, the rock is a siltstone. If the grains are not distinguishable with a hand lens, the rock is a claystone.

FIGURE 4

GRAIN SIZE CLASSIFICATION FOR ROCKS

Particle Name	Grain Size Diameter
Cobbles	> 64 mm
Pebbles	4 - 64 mm
Granules	2 - 4 mm
Very Coarse Sand	1 - 2 mm
Coarse Sand	0.5 - 1 mm
Medium Sand	0.25 - 0.5 mm
Fine Sand	0.125 - 0.25 mm
Very Fine Sand	0.0625 - 0.125 mm
Silt	0.0039 - 0.0625 mm

After Wentworth, 1922

5.3.2 Color

The color of a rock can be determined in a similar manner as for soil samples. Rock core samples shall be classified while wet, when possible, and air cored samples shall be scraped clean of cuttings prior to color classifications.

Rock color charts shall not be used unless specified by the Project Manager.

5.3.3 Bedding Thickness

The bedding thickness designations applied to soil classification (see Figure 3) will also be used for rock classification.

5.3.4 Hardness

The hardness of a rock is a function of the compaction, cementation, and mineralogical composition of the rock. A relative scale for sedimentary rock hardness is as follows:

- Soft - Weathered, considerable erosion of core, easily gouged by screwdriver, scratched by fingernail. Soft rock crushes or deforms under pressure of a pressed hammer. This term is always used for the hardness of the saprolite (decomposed rock which occupies the zone between the lowest soil horizon and firm bedrock).
- Medium soft - Slight erosion of core, slightly gouged by screwdriver, or breaks with crumbly edges from single hammer blow.
- Medium hard - No core erosion, easily scratched by screwdriver, or breaks with sharp edges from single hammer blow.
- Hard - Requires several hammer blows to break and has sharp conchoidal breaks. Cannot be scratched with screwdriver.

Note the difference in usage here of the works "scratch" and "gouge." A scratch shall be considered a slight depression in the rock (do not mistake the scraping off of rock flour from drilling with a scratch in the rock itself), while a gouge is much deeper.

5.3.5 Fracturing

The degree of fracturing or brokenness of a rock is described by measuring the fractures or joint spacing. After eliminating drilling breaks, the average spacing is calculated and the fracturing is described by the following terms:

- Very broken (V. BR.) - Less than 2-inch spacing between fractures
- Broken (BR.) - 2-inch to 1-foot spacing between fractures
- Blocky (BL.) - 1- to 3-foot spacing between fractures
- Massive (M.) - 3 to 10-foot spacing between fractures

The structural integrity of the rock can be approximated by calculating the Rock Quality Designation (RQD) of cores recovered. The RQD is determined by adding the total lengths of all pieces exceeding 4 inches and dividing by the total length of the coring run, to obtain a percentage.

Method of Calculating RQD
(After Deere, 1964)

$$RQD\ \% = r/l \times 100$$

r = Total length of all pieces of the lithologic unit being measured, which are greater than 4 inches length, and have resulted from natural breaks. Natural breaks include slickensides, joints, compaction slicks, bedding plane partings (not caused by drilling), friable zones, etc.

l = Total length of the coring run.

5.3.6 Weathering

The degree of weathering is a significant parameter that is important in determining weathering profiles and is also useful in engineering designs. The following terms can be applied to distinguish the degree of weathering:

- Fresh - Rock shows little or no weathering effect. Fractures or joints have little or no staining and rock has a bright appearance.
- Slight - Rock has some staining which may penetrate several centimeters into the rock. Clay filling of joints may occur. Feldspar grains may show some alteration.
- Moderate - Most of the rock, with exception of quartz grains, is stained. Rock is weakened due to weathering and can be easily broken with hammer.
- Severe - All rock including quartz grains is stained. Some of the rock is weathered to the extent of becoming a soil. Rock is very weak.

5.3.7 Other Characteristics

The following items shall be included in the rock description:

- Description of contact between two rock units. These can be sharp or gradational.
- Stratification (parallel, cross stratified).
- Description of any filled cavities or vugs.
- Cementation (calcareous, siliceous, hematitic).
- Description of any joints or open fractures.
- Observation of the presence of fossils.
- Notation of joints with depth, approximate angle to horizontal, any mineral filling or coating, and degree of weathering.

All information shown on the boring logs shall be neat to the point where it can be reproduced on a copy machine for report presentation. The data shall be kept current to provide control of the drilling program and to indicate various areas requiring special consideration and sampling.

5.3.8 Additional Terms Used in the Description of Rock

The following terms are used to further identify rocks:

- Seam - Thin (12 inches or less), probably continuous layer.
- Some - Indicates significant (15 to 40 percent) amounts of the accessory material. For example, rock composed of seams of sandstone (70 percent) and shale (30 percent) would be "sandstone – some shale seams."
- Few - Indicates insignificant (0 to 15 percent) amounts of the accessory material. For example, rock composed of seam of sandstone (90 percent) and shale (10 percent) would be "sandstone – few shale seams."
- Interbedded - Used to indicate thin or very thin alternating seams of material occurring in approximately equal amounts. For example, rock composed of thin alternating seams of sandstone (50 percent) and shale (50 percent) would be "interbedded sandstone and shale."
- Interlayered - Used to indicate thick alternating seams of material occurring in approximately equal amounts.

The preceding sections describe the classification of sedimentary rocks. The following are some basic names that are applied to igneous rocks:

- Basalt - A fine-grained extrusive rock composed primarily of calcic plagioclase and pyroxene.
- Rhyolite - A fine-grained volcanic rock containing abundant quartz and orthoclase. The fine-grained equivalent of a granite.
- Granite - A coarse-grained plutonic rock consisting essentially of alkali feldspar and quartz.
- Diorite - A coarse-grained plutonic rock consisting essentially of sodic plagioclase and hornblende.
- Gabbro - A coarse-grained plutonic rock consisting of calcic plagioclase and clinopyroxene. Loosely used for any coarse-grained dark igneous rock.

The following are some basic names that are applied to metamorphic rocks:

- Slate - A very fine-grained foliated rock possessing a well developed slaty cleavage. Contains predominantly chlorite, mica, quartz, and sericite.
- Phyllite - A fine-grained foliated rock that splits into thin flaky sheets with a silky sheen on cleavage surface.
- Schist - A medium to coarse-grained foliated rock with subparallel arrangement of the micaceous minerals which dominate its composition.
- Gneiss - A coarse-grained foliated rock with bands rich in granular and platy minerals.
- Quartzite - A fine- to coarse-grained nonfoliated rock breaking across grains, consisting essentially of quartz sand with silica cement.

5.4 Abbreviations

Abbreviations may be used in the description of a rock or soil. However, they shall be kept at a minimum. Following are some of the abbreviations that may be used:

C	-	Coarse	Lt	-	Light	Yl	-	Yellow
Med	-	Medium	BR	-	Broken	Or	-	Orange
F	-	Fine	BL	-	Blocky	SS	-	Sandstone
V	-	Very	M	-	Massive	Sh	-	Shale
Sl	-	Slight	Br	-	Brown	LS	-	Limestone
Occ	-	Occasional	Bl	-	Black	Fgr	-	Fine-grained
Tr	-	Trace						

5.5 Boring Logs and Documentation

This section describes in more detail the procedures to be used in completing boring logs in the field. Information obtained from the preceding sections shall be used to complete the logs. A sample boring log has been provided as Figure 5.

The field geologist/engineer shall use this example as a guide in completing each boring log. Each boring log shall be fully described by the geologist/engineer as the boring is being drilled. Every sheet contains space for 25 feet of log. Information regarding classification details is provided either on the back of the boring log or on a separate sheet, for field use.

5.5.1 Soil Classification

- Identify site name, boring number, job number, etc. Elevations and water level data to be entered when surveyed data is available.

- Enter sample number (from SPT) under appropriate column. Enter depth sample was taken from (1 block = 1 foot). Fractional footages, i.e., change of lithology at 13.7 feet, shall be lined off at the proportional location between the 13- and 14-foot marks. Enter blow counts (Standard Penetration Resistance) diagonally (as shown). Standard penetration resistance is covered in Section 5.2.3.

- Determine sample recovery/sample length as shown. Measure the total length of sample recovered from the split-spoon sampler, including material in the drive shoe. Do not include cuttings or wash material that may be in the upper portion of the sample tube.

- Indicate any change in lithology by drawing a line at the appropriate depth. For example, if clayey silt was encountered from 0 to 5.5 feet and shale from 5.5 to 6.0 feet, a line shall be drawn at this increment. This information is helpful in the construction of cross-sections. As an alternative, symbols may be used to identify each change in lithology.

- The density of granular soils is obtained by adding the number of blows for the last two increments. Refer to Density of Granular Soils Chart on back of log sheet. For consistency of cohesive soils refer also to the back of log sheet - Consistency of Cohesive Soils. Enter this information under the appropriate column. Refer to Section 5.2.3.

FIGURE 5
COMPLETED BORING LOG (EXAMPLE)

BOREHOLE LOG

Page 1 of 1

PROJECT NAME: NSB - SITE
PROJECT NUMBER: 9594
DRILLING COMPANY: SOILTEST CO.
DRILLING RIG: CME - 55
BORING NUMBER: SB/MW 1
DATE: 3/8/96
GEOLOGIST: S.J CONTI
DRILLER: R. ROCK

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	Color	Material Classification	USCS	Remarks	PID/FID Reading (ppm): Sample	Sampler B.Z	Borehole**	Drillier B.Z**
S-1	0.0	7/6	1.5/2.0		M DENSE	BRN TO BLK	SILTY SAND - SOME ROCK FR; TR BRICKS (FILL)	SM	MOIST SL. ORG. ODOR FILL TO 4'±	5	10	10	10
C 0800	2.0	9/10											
	4.0			4.0									
S-2 C		5/7	2.9/2.0		M DENSE	BRN	SILTY SAND - TR FINE GRAVEL	SM	MOIST - W ODOR NAT. MATL. TOOK SAMPLE SB01-0406 FOR ANALYSIS	10	10	-	-
0810	6.0	9/8											
	•			7' ∇									
	8.0			8.0									
S-3 C		6/8	1.9/2.0		DENSE	TAN BRN	FINE TO COARSE SAND TR F. GRAVEL	SW	WET HIT WATER @ 7'±	0	0	0	0
0820	10.0	17/16											
	12.0			12.0									
S-4 C		7/6	1.6/2.0		STIFF	GRAY	SILTY CLAY	CL	MOIST → WET	0	.5	-	-
0830	14.0	5/8											
	15.0			15.0					AUGER REF @ 15'				
				11:15 HRS	M HARD	BRN	SILTSTONE	VBR	WEATHERED				
				16					LD & JNTS @ 15.5				
9/5	①		4.0/5.0						WATER STAINS @ 16.5, 17.1, 17.5	0	0	0	0
				19'					LOSING SOME				
	20.0				HARD	GRAY	SANDSTONE - SOME SILTSTONE	BR	DRILL H20 @ 17'±				
									SET TEMP 6" CAS TO 15.5				
4.9/5.0	②		5.0/5.0						SET 2" Ø PVC SCREEN 16-25	0	0	0	0
									SAND 14-25				
	25.0			25					PELLETS 12-14				

* When rock coring, enter rock brokeness.
** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated response read.

• 1-20'
1-80'

Drilling Area Background (ppm): 0

Remarks: CME-55 R16, 4¼" ID HSA - 9" OD ±
2" SPLIT SPOONS - 140 LB HAMMER - 30" DROP
NX CORE IN BEDROCK RUN ① = 25 min. RUN ② = 15 min

Converted to Well: Yes ✓ No _____ Well I.D. #: MW-1

- Enter color of the material in the appropriate column.
- Describe material using the USCS. Limit this column for sample description only. The predominant material is described last. If the primary soil is silt but has fines (clay) - use clayey silt. Limit soil descriptors to the following:
  - Trace: 0 - 10 percent
  - Some: 11 - 30 percent
  - And/Or: 31 - 50 percent
- Also indicate under Material Classification if the material is fill or natural soils. Indicate roots, organic material, etc.
- Enter USCS symbol - use chart on back of boring log as a guide. If the soils fall into one of two basic groups, a borderline symbol may be used with the two symbols separated by a slash. For example ML/CL or SM/SP.
- The following information shall be entered under the "Remarks" column and shall include, but is not limited by, the following:
  - Moisture - estimate moisture content using the following terms - dry, moist, wet and saturated. These terms are determined by the individual. Whatever method is used to determine moisture, be consistent throughout the log.
  - Angularity - describe angularity of coarse grained particles using the terms angular, subangular, subrounded, or rounded. Refer to ASTM D 2488 or Earth Manual for criteria for these terms.
  - Particle shape - flat, elongated, or flat and elongated.
  - Maximum particle size or dimension.
  - Water level observations.
  - Reaction with HCl - none, weak, or strong.
- Additional comments:
  - Indicate presence of mica, caving of hole, when water was encountered, difficulty in drilling, loss or gain of water.
  - Indicate odor and Photoionization Detector (PID) or Flame Ionization Detector (FID) reading if applicable.
  - Indicate any change in lithology by drawing a line through the lithology change column and indicate the depth. This will help when cross-sections are subsequently constructed.
  - At the bottom of the page indicate type of rig, drilling method, hammer size and drop, and any other useful information (i.e., borehole size, casing set, changes in drilling method).

- Vertical lines shall be drawn (as shown in Figure 5) in columns 6 to 8 from the bottom of each sample to the top of the next sample to indicate consistency of material from sample to sample, if the material is consistent. Horizontal lines shall be drawn if there is a change in lithology, then vertical lines drawn to that point.

- Indicate screened interval of well, as needed, in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.2 Rock Classification

- Indicate depth at which coring began by drawing a line at the appropriate depth. Indicate core run depths by drawing coring run lines (as shown) under the first and fourth columns on the log sheet. Indicate RQD, core run number, RQD percent, and core recovery under the appropriate columns.

- Indicate lithology change by drawing a line at the appropriate depth as explained in Section 5.5.1.

- Rock hardness is entered under designated column using terms as described on the back of the log or as explained earlier in this section.

- Enter color as determined while the core sample is wet; if the sample is cored by air, the core shall be scraped clean prior to describing color.

- Enter rock type based on sedimentary, igneous or metamorphic. For sedimentary rocks use terms as described in Section 5.3. Again, be consistent in classification. Use modifiers and additional terms as needed. For igneous and metamorphic rock types use terms as described in Sections 5.3.8.

- Enter brokenness of rock or degree of fracturing under the appropriate column using symbols VBR, BR, BL, or M as explained in Section 5.3.5 and as noted on the back of the Boring Log.

- The following information shall be entered under the remarks column. Items shall include but are not limited to the following:

  - Indicate depths of joints, fractures and breaks and also approximate to horizontal angle (such as high, low), i.e., 70° angle from horizontal, high angle.
  - Indicate calcareous zones, description of any cavities or vugs.
  - Indicate any loss or gain of drill water.
  - Indicate drop of drill tools or change in color of drill water.

- Remarks at the bottom of Boring Log shall include:

  - Type and size of core obtained.
  - Depth casing was set.
  - Type of rig used.

- As a final check the boring log shall include the following:

  - Vertical lines shall be drawn as explained for soil classification to indicate consistency of bedrock material.

  - If applicable, indicate screened interval in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.5.3 Classification of Soil and Rock from Drill Cuttings

The previous sections describe procedures for classifying soil and rock samples when cores are obtained. However, some drilling methods (air/mud rotary) may require classification and borehole logging based on identifying drill cuttings removed from the borehole. Such cuttings provide only general information on subsurface lithology. Some procedures that shall be followed when logging cuttings are:

- Obtain cutting samples at approximately 5-foot intervals, sieve the cuttings (if mud rotary drilling) to obtain a cleaner sample, place the sample into a small sample bottle or "zip lock" bag for future reference, and label the jar or bag (i.e. hole number, depth, date, etc.). Cuttings shall be closely examined to determine general lithology.
- Note any change in color of drilling fluid or cuttings, to estimate changes in lithology.
- Note drop or chattering of drilling tools or a change in the rate of drilling, to determine fracture locations or lithologic changes.
- Observe loss or gain of drilling fluids or air (if air rotary methods are used), to identify potential fracture zones.
- Record this and any other useful information onto the boring log as provided in Figure 1.

This logging provides a general description of subsurface lithology and adequate information can be obtained through careful observation of the drilling process. It is recommended that split-barrel and rock core sampling methods be used at selected boring locations during the field investigation to provide detailed information to supplement the less detailed data generated through borings drilled using air/mud rotary methods.

5.6 Review

Upon completion of the borings logs, copies shall be made and reviewed. Items to be reviewed include:

- Checking for consistency of all logs.
- Checking for conformance to the guideline.
- Checking to see that all information is entered in their respective columns and spaces.

6.0 REFERENCES

Unified Soil Classification System (USCS).

ASTM D2488, 1985.

Earth Manual, U.S. Department of the Interior, 1974.

7.0 RECORDS

Originals of the boring logs shall be retained in the project files.

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		Applicability Tetra Tech NUS, Inc.	
		Prepared Health & Safety	
Subject UTILITY LOCATING AND EXCAVATION CLEARANCE		Approved D. Senovich	

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1.0 PURPOSE

Utilities such as electric service lines, natural or propane gas lines, water and sewage lines, telecommunications, and steam lines are very often in the immediate vicinity of work locations. Contact with underground or overhead utilities can have serious consequences including employee injury/fatality, property and equipment damage, substantial financial impacts, and loss of utility service to users.

The purpose of this procedure is to provide minimum requirements and technical guidelines regarding the appropriate procedures to be followed when performing subsurface and overhead utility locating services. It is the policy of Tetra Tech NUS, Inc. (TtNUS) to provide a safe and healthful work environment for the protection of our employees. The purpose of this Standard Operating Procedure (SOP) is to aid in achieving the objectives of this policy, to present the acceptable procedures pertaining to utility locating and excavation clearance activities, and to present requirements and restrictions relevant to these types of activities. This SOP must be reviewed by any employee potentially involved with underground or overhead utility locating and avoidance activities.

2.0 SCOPE

This procedure applies to all TtNUS field activities where there may be potential contact with underground or overhead utilities. This procedure provides a description of the principles of operation, instrumentation, applicability, and implementability of typical methods used to determine the presence and avoidance of contact with utility services. This procedure is intended to assist with work planning and scheduling, resource planning, field implementation, and subcontractor procurement. Utility locating and excavation clearance requires site-specific information prior to the initiation of any such activities on a specific project. This SOP is not intended to provide a detailed description of methodology and instrument operation. Specialized expertise during both planning and execution of several of the methods presented may also be required.

3.0 GLOSSARY

Electromagnetic Induction (EMI) Survey - A geophysical exploration method whereby electromagnetic fields are induced in the ground and the resultant secondary electromagnetic fields are detected as a measure of ground conductivity.

Magnetometer – A device used for precise and sensitive measurements of magnetic fields.

Magnetic Survey – A geophysical survey method that depends on detection of magnetic anomalies caused by the presence of buried ferromagnetic objects.

Metal Detection – A geophysical survey method that is based on electromagnetic coupling caused by underground conductive objects.

Vertical Gradiometer – A magnetometer equipped with two sensors that are vertically separated by a fixed distance. It is best suited to map near surface features and is less susceptible to deep geologic features.

Ground Penetrating Radar – Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture.

4.0 RESPONSIBILITIES

Project Manager (PM)/Task Order Manager (TOM) - Responsible for ensuring that all field activities are conducted in accordance with this procedure.

Site Manager (SM)/Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved SOPs or as otherwise directed by the approved project plan(s).

Site Health & Safety Officer (SHSO) – Responsible to provide technical assistance and verify full compliance with this SOP. The SHSO is also responsible for reporting any deficiencies to the Corporate Health and Safety Manager (HSM) and to the PM/TOM.

Health & Safety Manager (HSM) – Responsible for preparing, implementing, and modifying corporate health and safety policy and this SOP.

Site Personnel – Responsible for performing their work activities in accordance with this SOP and the TtNUS Health and Safety Policy.

5.0 PROCEDURES

This procedure addresses the requirements and technical procedures that must be performed to minimize the potential for contact with underground and overhead utility services. These procedures are addressed individually from a buried and overhead standpoint.

5.1 Buried Utilities

Buried utilities present a heightened concern because their location is not typically obvious by visual observation, and it is common that their presence and/or location is unknown or incorrectly known on client properties. This procedure must be followed prior to beginning any subsurface probing or excavation that might potentially be in the vicinity of underground utility services. In addition, the Utility Clearance Form (Attachment 3) must be completed for every location or cluster of locations where intrusive activities will occur.

Where the positive identification and de-energizing of underground utilities cannot be obtained and confirmed using the following steps, the PM/TOM is responsible for arranging for the procurement of a qualified, experienced, utility locating subcontractor who will accomplish the utility location and demarcation duties specified herein.

1. A comprehensive review must be made of any available property maps, blue lines, or as-builts prior to site activities. Interviews with local personnel familiar with the area should be performed to provide additional information concerning the location of potential underground utilities. Information regarding utility locations shall be added to project maps upon completion of this exercise.

2. A visual site inspection must be performed to compare the site plan information to actual field conditions. Any findings must be documented and the site plan/maps revised. The area(s) of proposed excavation or other subsurface activities must be marked at the site in white paint or pin flags to identify those locations of the proposed intrusive activities. The site inspection should focus on locating surface indications of potential underground utilities. Items of interest include the presence of nearby area lights, telephone service, drainage grates, fire hydrants, electrical service vaults/panels, asphalt/concrete scares and patches, and topographical depressions. Note the location of any emergency shut off switches. Any additional information regarding utility

locations shall be added to project maps upon completion of this exercise and returned to the PM/TOM.

3. If the planned work is to be conducted on private property (e.g., military installations, manufacturing facilities, etc.) the FOL must identify and contact appropriate facility personnel (e.g., public works or facility engineering) before any intrusive work begins to inquire about (and comply with) property owner requirements. It is important to note that private property owners may require several days to several weeks advance notice prior to locating utilities.

4. If the work location is on public property, the state agency that performs utility clearances must be notified (see Attachment 1). State "one-call" services must be notified prior to commencing fieldwork per their requirements. Most one-call services require, by law, 48- to 72-hour advance notice prior to beginning any excavation. Such services typically assign a "ticket" number to the particular site. This ticket number must be recorded for future reference and is valid for a specific period of time, but may be extended by contacting the service again. The utility service will notify utility representatives who then mark their respective lines within the specified time frame. It should be noted that most military installations own their own utilities but may lease service and maintenance from area providers. Given this situation, "one call" systems may still be required to provide location services on military installations.

5. Utilities must be identified and their locations plainly marked using pin flags, spray paint, or other accepted means. The location of all utilities must be noted on a field sketch for future inclusion on project maps. Utility locations are to be identified using the following industry-standard color code scheme, unless the property owner or utility locator service uses a different color code:

white	excavation/subsurface investigation location
red	electrical
yellow	gas, oil, steam
orange	telephone, communications
blue	water, irrigation, slurry
green	sewer, drain

6. Where utility locations are not confirmed with a high degree of confidence through drawings, schematics, location services, etc., the work area must be thoroughly investigated prior to beginning the excavation. In these situations, utilities must be identified using safe and effective methods such as passive and intrusive surveys, or the use of non-conductive hand tools. Also, in situations where such hand tools are used, they should always be used in conjunction with suitable detection equipment, such as the items described in Section 6.0 of this SOP. Each method has advantages and disadvantages including complexity, applicability, and price. It also should be noted that in some states, initial excavation is required by hand to a specified depth.

7. At each location where trenching or excavating will occur using a backhoe or other heavy equipment, and where utility identifications and locations cannot be confirmed prior to groundbreaking, the soil must be probed using a device such as a tile probe which is made of non-conductive material such as fiberglass. If these efforts are not successful in clearing the excavation area of suspect utilities, hand shoveling must be performed for the perimeter of the intended excavation.

8. All utilities uncovered or undermined during excavation must be structurally supported to prevent potential damage. Unless necessary as an emergency corrective measure, TtNUS shall not make any repairs or modifications to existing utility lines without prior permission of the utility owner, property owner, and Corporate HSM. All repairs require that the line be locked-out/tagged-out prior to work.

5.2 Overhead Power Lines

If it is necessary to work within the minimum clearance distance of an overhead power line, the overhead line must be de-energized and grounded, or re-routed by the utility company or a registered electrician. If protective measures such as guarding, isolating, or insulating are provided, these precautions must be adequate to prevent employees from contacting such lines directly with any part of their body or indirectly though conductive materials, tools, or equipment.

The following table provides the required minimum clearances for working in proximity to overhead power lines.

Nominal Voltage	Minimum Clearance
0 -50 kV	10 feet, or one mast length; whichever is greater
50+ kV	10 feet plus 4 inches for every 10 kV over 50 kV or 1.5 mast lengths; whichever is greater

6.0 UNDERGROUND LOCATING TECHNIQUES

A variety of supplemental utility locating approaches are available and can be applied when additional assurance is needed. The selection of the appropriate method(s) to employ is site-specific and should be tailored to the anticipated conditions, site and project constraints, and personnel capabilities.

6.1 Geophysical Methods

Geophysical methods include electromagnetic induction, magnetics, and ground penetrating radar. Additional details concerning the design and implementation of electromagnetic induction, magnetics, and ground penetrating radar surveys can be found in one or more of the TtNUS SOPs included in the References (Section 8.0).

Electromagnetic Induction

Electromagnetic Induction (EMI) line locators operate either by locating a background signal or by locating a signal introduced into the utility line using a transmitter. A utility line acts like a radio antenna, producing electrons, which can be picked up with a radiofrequency receiver. Electrical current carrying conductors have a 60HZ signal associated with them. This signal occurs in all power lines regardless of voltage. Utilities in close proximity to power lines or used as grounds may also have a 60HZ signal, which can be picked up with an EM receiver. A typical example of this type of geophysical equipment is an EM-61.

EMI locators specifically designed for utility locating use a special signal that is either indirectly induced onto a utility line by placing the transmitter above the line or directly induced using an induction clamp. The clamp induces a signal on the specific utility and is the preferred method of tracing since there is little chance of the resulting signals being interfered with. A good example of this type of equipment is the Schonstedt® MAC-51B locator. The MAC-51B performs inductively traced surveys, simple magnetic locating, and traced nonmetallic surveys.

When access can be gained inside a conduit to be traced, a flexible insulated trace wire can be used. This is very useful for non-metallic conduits but is limited by the availability of gaining access inside the pipe.

Magnetics

Magnetic locators operate by detecting the relative amounts of buried ferrous metal. They are incapable of locating or identifying nonferrous utility lines but can be very useful for locating underground storage tanks (UST's), steel utility lines, and buried electrical lines. A typical example of this type of equipment is the Schonstedt® GA-52Cx locator. The GA-52Cx is capable of locating 4-inch steel pipe up to 8 feet deep.

Non-ferrous lines are often located by using a typical plumbing tool (snake) fed through the line. A signal is then introduced to the snake that is then traced.

Ground Penetrating Radar

Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture. In general, an object which is harder than the surrounding soil will reflect a stronger signal. Utilities, tunnels, UST's, and footings will reflect a stronger signal than the surrounding soil. Although this surface detection method may determine the location of a utility, this method does not specifically identify utilities (i.e., water vs. gas, electrical vs. telephone); hence, verification may be necessary using other methods. This method is somewhat limited when used in areas with clay soil types or with a high water table.

6.2 Passive Detection Surveys

Acoustic Surveys

Acoustic location methods are generally most applicable to waterlines or gas lines. A highly sensitive Acoustic Receiver listens for background sounds of water flowing (at joints, leaks, etc.) or to sounds introduced into the water main using a transducer. Acoustics may also be applicable to determine the location of plastic gas lines.

Thermal Imaging

Thermal (i.e., infrared) imaging is a passive method for detecting the heat emitted by an object. Electronics in the infrared camera convert subtle heat differentials into a visual image on the viewfinder or a monitor. The operator does not look for an exact temperature; rather they look for heat anomalies (either elevated or suppressed temperatures) characteristic of a potential utility line.

The thermal fingerprint of underground utilities results from differences in temperature between the atmosphere and the fluid present in a pipe or the heat generated by electrical resistance. In addition, infrared scanners may be capable of detecting differences in the compaction, temperature and moisture content of underground utility trenches. High-performance thermal imagery can detect temperature differences to hundredths of a degree.

6.3 Intrusive Detection Surveys

Vacuum Excavation

Vacuum excavation is used to physically expose utility services. The process involves removing the surface material over approximately a 1' x 1' area at the site location. The air-vacuum process proceeds with the simultaneous action of compressed air-jets to loosen soil and vacuum extraction of the resulting

debris. This process ensures the integrity of the utility line during the excavation process, as no hammers, blades, or heavy mechanical equipment comes into contact with the utility line, eliminating the risk of damage to utilities. The process continues until the utility is uncovered. Vacuum excavation can be used at the proposed site location to excavate below the "utility window" which is usually 8 feet.

Hand Excavation

When the identification and location of underground utilities cannot be positively confirmed through document reviews and/or other methods, borings and excavations may be cleared via the use of non-conductive hand tools. This should always be done in conjunction with the use of detection equipment. This would be required for all locations where there is a potential to impact buried utilities. The minimum hand-excavation depth that must be reached is to be determined considering the geographical location of the work site. This approach recognizes that the placement of buried utilities is influenced by frost line depths that vary by geographical region. Attachment 2 presents frost line depths for the regions of the contiguous United States. At a minimum, hand excavation depths must be at least to the frost line depth (see Attachment 2) plus two (2) feet, but never less than 4 feet below ground surface (bgs). For hand excavation, the hole created must be reamed large enough to be at least the diameter of the drill rig auger or bit prior to drilling. For soil gas surveys, the survey probe shall be placed as close as possible to the cleared hand excavation. It is important to note that a post-hole digger must not be used in this type of hand excavation activity.

Tile Probe Surveys

For some soil types, site conditions, and excavation requirements, non-conductive tile probes may be used. A tile probe is a "T"-handled rod of varying lengths that can be pushed into the soil to determine if any obstructions exist at that location. Tile probes constructed of fiberglass or other nonconductive material are readily-available from numerous vendors. Tile probes must be performed to the same depth requirements as previously specified. As with other types of hand excavating activities, the use of a non-conductive tile probe, should always be in conjunction with suitable utility locating detection equipment.

7.0 INTRUSIVE ACTIVITIES SUMMARY

The following list summarizes the activities that must be performed prior to beginning subsurface activities:

1. Map and mark all subsurface locations and excavation boundaries using white paint or markers specified by the client or property owner.

2. Notify the property owner and/or client that the locations are marked. At this point, drawings of locations or excavation boundaries shall be provided to the property owner and/or client so they may initiate (if applicable) utility clearance.

   Note: Drawings with confirmed locations should be provided to the property owner and/or client as soon as possible to reduce potential time delays.

3. Notify "One Call" service. If possible, arrange for an appointment to show the One Call representative the surface locations or excavation boundaries in person. This will provide a better location designation to the utilities they represent. You should have additional drawings should you need to provide plot plans to the One Call service.

4. Implement supplemental utility detection techniques as necessary and appropriate to conform utility locations or the absence thereof.

5. Complete Attachment 3, Utility Clearance Form. This form should be completed for each excavation location. In situations where multiple subsurface locations exist within the close proximity of one another, one form may be used for multiple locations provided those locations are noted on the Utility Clearance Form. Upon completion, the Utility Clearance Form and revised/annotated utility location map becomes part of the project file.

8.0 REFERENCES

OSHA Letter of Interpretation, Mr. Joseph Caldwell, Attachment 4
OSHA 29 CFR 1926(b)(2)
OSHA 29 CFR 1926(b)(3)
TtNUS Utility Locating and Clearance Policy
TtNUS SOP GH-3.1; Resistivity and Electromagnetic Induction
TtNUS SOP GH-3.2; Magnetic and Metal Detection Surveys
TtNUS SOP GH-3.4; Ground-penetrating Radar Surveys

ATTACHMENT 1
LISTING OF UNDERGROUND UTILITY CLEARANCE RESOURCES

American Public Works Association
2345 Grand Boulevard, Suite 500, Kansas City, MO 64108-2625
Phone (816) 472-6100 • Fax (816) 472-1610
Web www.apwa.net • E-mail apwa@apwa.net

ONE-CALL SYSTEMS INTERNATIONAL CONDENSED DIRECTORY

Alabama
Alabama One-Call
1-800-292-8525

Alaska
Locate Call Center of Alaska, Inc.
1-800-478-3121

Arizona
Arizona Blue Stake
1-800-782-5348

Arkansas
Arkansas One Call System, Inc.
1-800-482-8998

California
Underground Service Alert North
1-800-227-2600
Underground Service Alert of Southern California
1-800-227-2600

Colorado
Utility Notification Center of Colorado
1-800-922-1987

Connecticut
Call Before You Dig
1-800-922-4455

Delaware
Miss Utility of Delmarva
1-800-282-8555

Florida
Sunshine State One-Call of Florida, Inc.
1-800-432-4770

Georgia
Underground Protection Center, Inc.
1-800-282-7411

Hawaii
Underground Service Alert North
1-800-227-2600

Idaho
Dig Line Inc.
1-800-342-1585
Kootenai County One-Call
1-800-428-4950
Shoshone - Benewah One-Call
1-800-398-3285

Illinois
JULIE, Inc.
1-800-892-0123
Digger (Chicago Utility Alert Network)
312-744-7000

Indiana
Indiana Underground Plant Protection Service
1-800-382-5544

Iowa
Iowa One-Call
1-800-292-8989

Kansas
Kansas One-Call System, Inc.
1-800-344-7233

Kentucky
Kentucky Underground Protection Inc.
1-800-752-6007

Louisiana
Louisiana One Call System, Inc.
1-800-272-3020

Maine
Dig Safe System, Inc.
1-888-344-7233

Maryland
Miss Utility
1-800-257-7777
Miss Utility of Delmarva
1-800-282-8555

Massachusetts
Dig Safe System, Inc.
1-888-344-7233

Michigan
Miss Dig System, Inc.
1-800-482-7171

Minnesota
Gopher State One Call
1-800-252-1166

Mississippi
Mississippi One-Call System, Inc
1-800-227-6477

Missouri
Missouri One-Call System, Inc.
1-800-344-7483

Montana
Utilities Underground Protection Center
1-800-424-5555
Montana One Call Center
1-800-551-8344

Nebraska
Diggers Hotline of Nebraska
1-800-331-5666

Nevada
Underground Service Alert North
1-800-227-2600

New Hampshire
Dig Safe System, Inc.
1-888-344-7233

New Jersey
New Jersey One Call
1-800-272-1000

New Mexico
New Mexico One Call System, Inc.
1-800-321-2537
Las Cruces- Dona Ana Blue Stakes
1-888-526-0400

New York
Dig Safely New York
1-800-962-7962
New York City- Long Island One Call Center
1-800-272-4480

North Carolina
The North Carolina One-Call Center, Inc.
1-800-632-4949

North Dakota
North Dakota One-Call
1-800-795-0555

Ohio
Ohio Utilities Protection Service
1-800-362-2764
Oil & Gas Producers Underground Protect'n Svc
1-800-925-0988

Oklahoma
Call Okie
1-800-522-6543

Oregon
Oregon Utility Notification Center/One Call Concepts
1-800-332-2344

Pennsylvania
Pennsylvania One Call System, Inc.
1-800-242-1776

Rhode Island
Dig Safe System, Inc.
1-888-344-7233

South Carolina
Palmetto Utility Protection Service Inc.
1-888-721-7877

South Dakota
South Dakota One Call
1-800-781-7474

Tennessee
Tennessee One-Call System, Inc.
1-800-351-1111

ATTACHMENT 1 (Continued)

Texas
Texas One Call System
1-800-245-4545
Texas Excavation Safety System, Inc.
1-800-344-8377
Lone Star Notification Center
1-800-669-8344

Utah
Blue Stakes of Utah
1-800-662-4111

Vermont
Dig Safe System, Inc.
1-888-344-7233

Virginia
Miss Utility of Virginia
1-800-552-7001
Miss Utility (Northern Virginia)
1-800-257-7777

Washington
Utilities Underground Location Center
1-800-424-5555
Northwest Utility Notification Center
1-800-553-4344
Inland Empire Utility Coordinating Council
509-456-8000

West Virginia
Miss Utility of West Virginia, Inc.
1-800-245-4848

Wisconsin
Diggers Hotline, Inc.
1-800-242-8511

Wyoming
Wyoming One-Call System, Inc.
1-800-348-1030
Call Before You Dig of Wyoming
1-800-849-2476

District of Columbia
Miss Utility
1-800-257-7777

Alberta
Alberta One-Call Corporation
1-800-242-3447

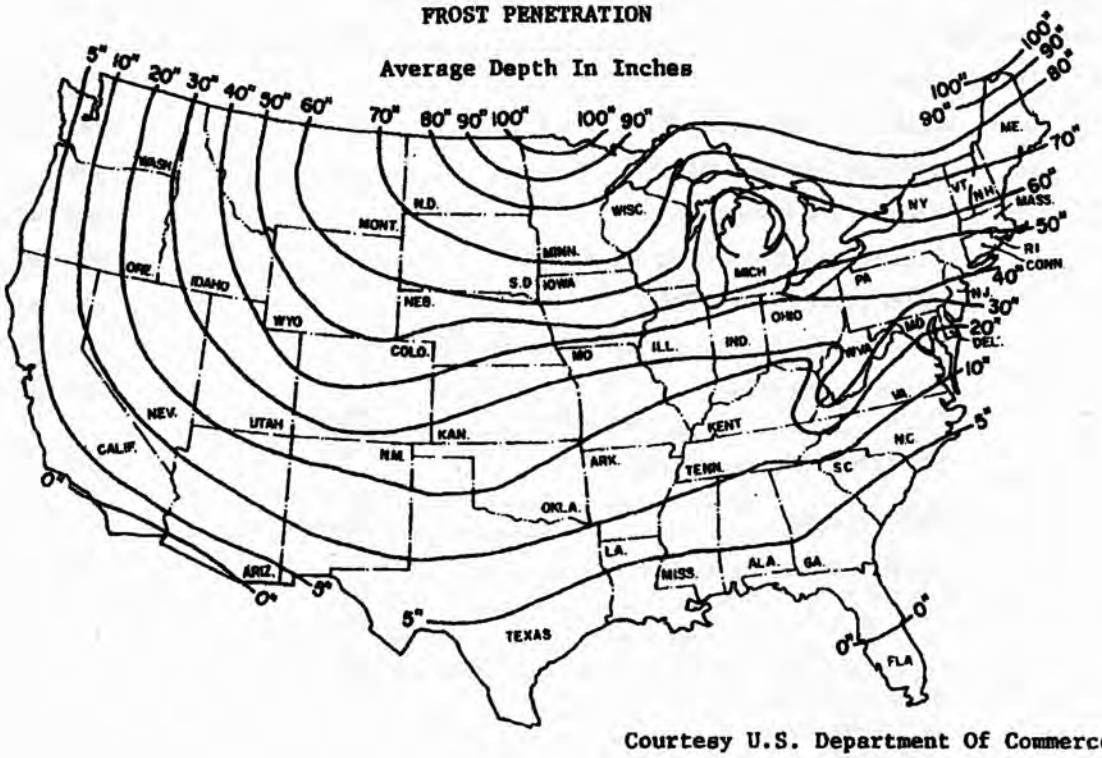
British Columbia
BC One Call
1-800-474-6886

Ontario
Ontario One-Call System
1-800-400-2255

Quebec
Info-Excavation
1-800-663-9228

ATTACHMENT 2

FROST LINE PENETRATION DEPTHS BY GEOGRAPHIC LOCATION



Courtesy U.S. Department Of Commerce

ATTACHMENT 3
UTILITY CLEARANCE FORM

Client: _____ Project Name: _____

Project No.: _____ Completed By: _____

Location Name: _____ Work Date: _____

Excavation Method/Overhead Equipment: _____

1. Underground Utilities — Circle One

a)	Review of existing maps?	yes no N/A
b)	Interview local personnel?	yes no N/A
c)	Site visit and inspection?	yes no N/A
d)	Excavation areas marked in the field?	yes no N/A
e)	Utilities located in the field?	yes no N/A
f)	Located utilities marked/added to site maps?	yes no N/A
g)	Client contact notified Name _____ Telephone: _____ Date: _____	yes no N/A
g)	State One-Call agency called? Caller: _____ Ticket Number: _____ Date: _____	yes no N/A
h)	Geophysical survey performed? Survey performed by: _____ Method: _____ Date: _____	yes no N/A
i)	Hand excavation performed (with concurrent use of utility detection device)? Completed by: _____ Total depth: _____ feet Date: _____	yes no N/A
j)	Trench/excavation probed? Probing completed by: _____ Depth/frequency: _____ Date: _____	yes no N/A

2. Overhead Utilities — Present Absent

a)	Determination of nominal voltage	yes no N/A
b)	Marked on site maps	yes no N/A
c)	Necessary to lockout/insulate/re-route	yes no N/A
d)	Document procedures used to lockout/insulate/re-route	yes no N/A
e)	Minimum acceptable clearance (SOP Section 5.2): _____	

3. Notes:

_____

_____

_____

Approval:

_____ _____

Site Manager/Field Operations Leader Date

c: PM/Project File
Program File

ATTACHMENT 4
OSHA LETTER OF INTERPRETATION

Mr. Joseph Caldwell
Consultant
Governmental Liaison
Pipeline Safety Regulations
211 Wilson Boulevard
Suite 700
Arlington, Virginia 22201

Re: Use of hydro-vacuum or non-conductive hand tools to locate underground utilities.

Dear Mr. Caldwell:

In a letter dated July 7, 2003, we responded to your inquiry of September 18, 2002, regarding the use of hydro-vacuum equipment to locate underground utilities by excavation. After our letter to you was posted on the OSHA website, we received numerous inquiries that make it apparent that aspects of our July 7 letter are being misunderstood. In addition, a number of industry stakeholders, including the National Utility Contractors Association (NUCA), have provided new information regarding equipment that is available for this work.

To clarify these issues, we are withdrawing our July 7 letter and issuing this replacement response to your inquiry.

Question: *Section 1926.651 contains several requirements that relate to the safety of employees engaged in excavation work. Specifically, paragraphs (b)(2) and (b)(3) relate in part to the safety of the means used to locate underground utility installations that, if damaged during an uncovering operation, could pose serious hazards to employees.*

Under these provisions, what constitutes an acceptable method of uncovering underground utility lines, and further, would the use of hydro-vacuum excavation be acceptable under the standard?

Answer

Background

Two sections of 29 CFR 1926 Subpart P (Excavations), 1926.651(Specific excavation requirements), govern methods for uncovering underground utility installations. Specifically, paragraph (b)(2) states:

> When utility companies or owners cannot respond to a request to locate underground utility installations within 24 hours * * * or cannot establish the exact location of these installations, the employer may proceed, provided the employer does so with caution, and provided detection equipment or other acceptable means to locate utility installations are used. (emphasis added).

Paragraph (b)(3) provides:

ATTACHMENT 4 (Continued)

> When excavation operations approach the estimated location of underground installations, the exact location of the installations shall be determined by safe and acceptable means. (emphasis added).

Therefore, "acceptable means" must be used where the location of the underground utilities have not been identified by the utility companies and detection equipment is not used.

Subpart P does not contain a definition of either "other acceptable means" or "safe and acceptable means." The preambles to both the proposed rule and the final rule discussed the rationale behind the wording at issue. For example, the preamble to the proposed rule, 52 Fed. Reg. 12301 (April 15, 1987), noted that a 1972 version of this standard contained language that specified "careful probing or hand digging" as the means to uncover utilities. The preamble then noted that an amendment to the 1972 standard later deleted that language "to allow other, *equally effective means* of locating such installations." The preamble continued that in the 1987 proposed rule, OSHA again proposed using language in section (b)(3) that would provide another example of an acceptable method of uncovering utilities that could be used *where the utilities have not been marked and detection equipment is not being used* – "probing with hand-held tools." This method was rejected in the final version of 29 CFR 1926. As OSHA explained in the preamble to the final rule, 54 Fed. Reg. 45916 (October 31, 1989):

> OSHA received two comments * * * and input from ACCSH [OSHA's Advisory Committee on Construction Safety and Health] * * * on this provision. All commenters recommended dropping 'such as probing with hand-held tools' from the proposed provision, because this could create a hazard to employees by damaging the installation or its insulation.

In other words, the commenters objected to the use of hand tools being used unless detection equipment was used in conjunction with them. OSHA then concluded its discussion relative to this provision by agreeing with the commentators and ultimately not including any examples of "acceptable means" in the final provision.

Non-conductive hand tools are permitted

This raises the question of whether the standard permits the use of hand tools alone -- without also using detection equipment. NUCA and other industry stakeholders have recently informed us that non-conductive hand tools that are appropriate to be used to locate underground utilities are now commonly available.

Such tools, such as a "shooter" (which has a non-conductive handle and a snub nose) and non-conductive or insulated probes were not discussed in the rulemaking. Since they were not considered at that time, they were not part of the class of equipment that was thought to be unsafe for this purpose. Therefore, we conclude that the use of these types of hand tools, when used with appropriate caution, is an "acceptable means" for locating underground utilities.

ATTACHMENT 4 (Continued)

Hydro-vacuum excavation

It is our understanding that some hydro-vacuum excavation equipment can be adjusted to use a minimum amount of water and suction pressure. When appropriately adjusted so that the equipment will not damage underground utilities (especially utilities that are particularly vulnerable to damage, such as electrical lines), use of such equipment would be considered a "acceptable means" of locating underground utilities. However, if the equipment cannot be sufficiently adjusted, then this method would not be acceptable under the standard.

Other technologies

We are not suggesting that these are the only devices that would be "acceptable means" under the standard. Industry stakeholders have informed us that there are other types of special excavation equipment designed for safely locating utilities as well.

We apologize for any confusion our July 7 letter may have caused. If you have further concerns or questions, please feel free to contact us again by fax at: U.S. Department of Labor, OSHA, Directorate of Construction, Office of Construction Standards and Compliance Assistance, fax # 202-693-1689. You can also contact us by mail at the above office, Room N3468, 200 Constitution Avenue, N.W., Washington, D.C. 20210, although there will be a delay in our receiving correspondence by mail.

Sincerely,

Russell B. Swanson, Director
Directorate of Construction

NOTE: OSHA requirements are set by statute, standards and regulations. Our interpretation letters explain these requirements and how they apply to particular circumstances, but they cannot create additional employer obligations. This letter constitutes OSHA=s interpretation of the requirements discussed. Note that our enforcement guidance may be affected by changes to OSHA rules. Also, from time to time we update our guidance in response to new information. To keep apprised of such developments, you can consult OSHA's website at http://www.osha.gov.

TETRA TECH	STANDARD OPERATING PROCEDURES	Number SA-1.2	Page 1 of 21
		Effective Date 03/28/2008	Revision 5
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject SURFACE WATER AND SEDIMENT SAMPLING		Approved Tom Johnston	

TABLE OF CONTENTS

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes procedures and equipment commonly used for collecting environmental samples of surface water and aquatic sediment for either onsite examination and chemical testing or for offsite laboratory analysis.

2.0 SCOPE

The information presented in this document is applicable to all environmental sampling of surface waters (Section 5.3) and aquatic sediments (Section 5.5), except where the analyte(s) may interact with the sampling equipment. The collection of concentrated sludges or hazardous waste samples from disposal or process lagoons often requires methods, precautions, and equipment different from those described herein.

3.0 GLOSSARY

Analyte – Chemical or radiochemical material whose concentration, activity, or mass is measured.

Composite Sample – A sample representing a physical average of grab samples.

Environmental Sample – A quantity of material collected in support of an environmental investigation that does not require special handling or transport considerations as detailed in SOP SA-6.1.

Grab Sample – A portion of material collected to represent material or conditions present at a single unit of space and time.

Hazardous Waste Sample – A sample containing (or suspected to contain) concentrations of contaminants that are high enough to require special handling and/or transport considerations per SOP SA-6.1.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, initial sampling locations, and field procedures used in the collection of soil samples. The Project Manager also has the overall responsibility for seeing that all surface water and sediment sampling activities are properly conducted by appropriately trained personnel in accordance with applicable planning documents.

Field Operations Leader - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that

custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection of surface water and sediment samples. The FOL is ultimately responsible for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan (HASP). This includes but is not be limited to performing air quality monitoring during sampling and boring and excavation activities, and ensuring that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO or SSO designee may also be required to advise the FOL on other safety-related matters regarding boring and sampling, such as mitigative measures to address potential hazards from hazardous objects or conditions.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP and other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, , container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (I.e., weather) conditions.
- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

5.0 HEALTH AND SAFETY

Precautions to preserve the health and safety of field personnel implementing this SOP are distributed throughout. The following general hazards may also exist during field activities, and the means of avoiding them must be used to preserve the health and safety of field personnel:

~~**Bridge/Boat Sampling** – Potential hazards associated with this activity include:~~

- ~~Traffic – one of the primary concerns as samplers move across a bridge because free space of travel is not often provided. Control measures should include:~~
  - ~~When sampling from a bridge, if the samplers do not have at least 6 feet of free travel space or physical barriers separating them and the traffic patterns, the HASP will include a Traffic Control Plan.~~
  - ~~The use of warning signs and high-visibility vests are required to warn oncoming traffic and to increase the visibility of sample personnel.~~
- ~~Slips, trips, and falls from elevated surfaces are a primary concern. Fall protection shall be worn when or if samplers must lean over a rail to obtain sample material. A Fall Protection Competent~~

~~Person (in accordance with Occupational safety and Health Administration [OSHA] fall protection standards) must be assigned to ensure that fall protection is appropriately and effectively employed~~

- ~~Water hazards/drowning~~ – if someone enters the water from an elevated surface (such as a ~~bridge or~~ dock) and ~~when~~ sampling from a boat. To minimize this potential, personnel shall wear ~~United~~ States Coast Guard (USCG)-approved floatation devices, and the sampling crew ~~must~~ also have on hand a Type IV Throwable Personal ~~Floatation~~ Device with at least 90 ~~feet~~ of 3/8-inch rope. See Section 5.5.2 of this SOP.

- Within the HASP, provisions ~~will~~ also be provided concerning the requirement of a Safe Vessel Certification or the ~~necessity~~ to conduct a boat inspection prior to use. In addition, the HASP shall also specify requirements as to whether the operator must be certified as a ~~commercial~~ boat operator and whether members of the sampling team must have a state-specific safe boating certification.

Entering Water to Collect Samples – Several hazards are associated with this activity and can be mitigated as follows:

- Personnel must wear a USCG-approved Floatation Device (selected and identified in the HASP). The SSO shall ensure that the device selected is in acceptable condition and suitable for the individual using it. This includes consideration of the weight of the individual.

- Lifelines shall be employed from a point on the shore. This activity will always be conducted with a Buddy. See Section 6.5.2.

- Personnel shall carry a probe to monitor the bottom ahead of them for drop offs or other associated hazards.

- The person in the water shall exercise caution concerning the path traveled so that the lifeline does not become entangled in underwater obstructions such as logs, branches, stumps, etc., thereby restricting its effectiveness in extracting the person from the water.

- Personnel shall not enter waters on foot in situations where natural hazards including alligators, snakes, as well as sharks, gars, and other predators within inland waterways may exist.

- In all cases, working along and/or entering the water during high currents or flood conditions shall be prohibited.

- Personnel shall not enter bodies of water where known debris exists that could result in injuries from cuts and lacerations.

Sampling in marshes or tidal areas in some instances can be accomplished using an all-terrain vehicle (ATV). This is not the primary recommended approach because the vehicle may become disabled, or weather conditions or tidal changes could result in environmental damage as well as loss of the vehicle. The primary approach is recommended to be on foot where minimal disturbance would occur. The same precautions specified above with regard to sediment disturbance apply as well as the previously described safety concerns associated with natural hazards. The natural hazards include alligators, bees (nests in dead falls and tree trunks), snakes, etc. In addition, moving through and over this terrain is difficult and could result in muscle strain and slips, trips, and falls. Common sense dictates that the sampler selects the most open accessible route over moderate terrain. Move slowly and deliberately through challenging terrain to minimize falls. Mud boots or other supportive PPE should be considered and specified in the HASP to permit samplers to move over soft terrain with the least amount of effort. In these situations, it is also recommended, as the terrain allows, that supplies be loaded and transported in a sled over the soft ground.

Working in these areas, also recognize the following hazards and means of protection against them:

Insects are also a primary concern. These include mosquitoes, ticks, spiders, bees, ants, etc. The HASP will identify those particular to your area. Typical preventative measures include:

- Use insect repellant. Approval of various repellants should be approved by the Project Chemist or Project Manager.
- Wearing light-colored clothing to control heat load due to excessive temperatures. In addition, it makes it easier to detect crawling insects on your clothing.
- Taping pants to boots to deny access. Again, this is recommended to control access to the skin by crawling insects. Consultation with the Project Health and Safety Officer SSO/Health and Safety Manager is recommended under extreme heat loads because this will create conditions of heat stress.
- Performing a body check to remove insects. The quicker you remove ticks, the less likely they will become attached and transfer bacteria to your bloodstream. Have your Buddy check areas inaccessible to yourself. This includes areas such as the upper back and between shoulder blades where it is difficult for you to examine and even more difficult for you to remove.

Safety Reminder

If you are allergic to bee or ant stings, it is especially critical that you carry your doctor-recommended antidote with you in these remote sampling locations due to the extended time required to extract incapacitated individuals as well as the effort required to extract them. In these scenarios, instruct your Buddy in the proper administration of the antidote. In all cases, if you have received a sting, administer the antidote regardless of the immediate reaction, evacuate, and seek medical attention as necessary. The FOL and/or SSO will determine when and if you may return to the field based on the extent of the immune response and hazards or potential hazards identified in these locations. To the FOL and SSO, this is a serious decision you have to make as to whether to take someone vulnerable to these hazards into a remote location where you may not be able to carry them out. Consider it wisely.

Poisonous Plants – To minimize the potential of encountering poisonous plants in the field, at least one member of the field team needs to have basic knowledge of what these plants look like so that they can be recognized, pointed out to other field personnel, and avoided if at all possible. If the field team cannot avoid contact and must move through an area where these plants exist, the level of personal protective equipment (PPE) shall include Tyvek coveralls and enhanced decontamination procedures for the removal of oils from the tooling and/or equipment.

Temperature-Related Stress – Excessively cold temperatures may result in cold stress, especially when entering the water either intentionally or by accident. Provisions for combating this hazard should be maintained at the sample location during this activity. Excessively hot temperatures may result in heat stress especially in scenarios where equipment is packed through the marsh.

Because all of these activities are conducted outside, electrical storms are a significant concern. The following measures will be incorporated to minimize this hazard:

- Where possible, utilize commercial warning systems and weather alerts to detect storms moving into the area.
- If on or in the water, get out of the water. Move to vehicles or preferably into enclosed buildings with plumbing and wiring.
- Where warning systems are not available, follow the 30/30 Rule (*if there are less than 30 seconds between thunder and lightning, go inside for at least 30 minutes after the last thunder).*

See Section 4.0 of the Health and Safety Guidance Manual (HSGM) for additional protective measures.

6.0 PROCEDURES

6.1 Introduction

Collecting a representative sample of surface water or sediment may be difficult because of water movement, stratification, or heterogeneous distribution of the targeted analytes. To collect representative samples, one must standardize sampling methods related to site selection, sampling frequency, sample collection, sampling devices, and sample handling, preservation, and identification. Regardless of quality control applied during laboratory analyses and subsequent scrutiny of analytical data packages, reported data are no better than the confidence that can be placed in the representativeness of the samples. Consult Appendix C for guidance on sampling that should be considered during project planning and that may be helpful to field personnel.

6.1.1 ~~Surface Water Sampling Equipment~~

~~The selection of sampling equipment depends on the site conditions and sample type to be acquired. In general, the most representative samples are obtained from mid-channel at a stream depth of 0.5 feet in a well-mixed stream; however, project-specific planning documents will address site-specific sampling requirements including sample collection points and sampling equipment. The most frequently used samplers include the following:~~

- ~~Peristaltic pump~~
- ~~Bailer~~
- ~~Dip sampler~~
- ~~Weighted bottle~~
- ~~Hand pump~~
- ~~Kemmerer~~
- ~~Depth-integrating sampler~~

~~The dip sampler and weighted-bottle sampler are used most often, and detailed discussions for these devices and the Kemmerer sampler are addressed subsequently in this section.~~

~~The criteria for selecting a sampler include:~~

1. ~~Disposability and/or easy decontamination.~~
2. ~~Inexpensive cost (if the item is to be disposed).~~
3. ~~Ease of operation.~~

4. Non-reactive/non-contaminating properties - Teflon-coated, glass, stainless-steel or polyvinyl chloride (PVC) sample chambers are preferred (in that order).

Measurements collected for each sample (grab or each aliquot collected for compositing) shall include but not be limited to:

- Specific conductance
- Temperature
- pH
- Dissolved oxygen

Sample measurements shall be conducted as soon as the sample is acquired. Measurement techniques described in SOP SA-1.1 shall be followed. All pertinent data and results shall be recorded in a field notebook or on sample log sheets (see Attachment A) or an equivalent electronic form(s). These analyses may be selected to provide information on water mixing/stratification and potential contamination. Various types of water bodies have differing potentials for mixing and stratification.

In general, the following equipment if necessary for obtaining surface water samples:

- Required sampling equipment, which may include a remote sampling pole, weighted bottle sampler, Kemmerer sampler, or other device.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.
- Required PPE as directed in the project-specific planning document, which may include:
  - Nitrile surgeon's or latex gloves (layered as necessary).
  - Safety glasses.
  - Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.

> ***Safety Reminder***
> The use of latex products may elicit an allergic reaction in some people. Should this occur, remove the latex gloves, treat for an allergic reaction, and seek medical attention as necessary.

  - Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).
  - Required decontamination equipment.
  - Required sample containers.
  - Sealable polyethylene bags (e.g., Ziploc® baggies).
  - Heavy-duty cooler.
  - Ice.

- Paper towels and garbage bags.
- Chain-of-custody records and custody seals.

Dip Sampling

Specific procedures for collecting a dip or grab sample of surface water can vary based on site-specific conditions (e.g., conditions near the shore and how closely a sampler can safely get to the shore). The general procedure for collecting a sample using a pole or directly from the water body is as follows:

1. If using a remote sampling pole, securely attach the appropriate sample container to a pole of sufficient length to reach the water to be sampled. Samples for volatile analysis should be collected first. Use PPE as described in the HASP. When sample containers are provided pre-preserved or if the pole cannot accommodate a particular sample container, use a dedicated, clean, unpreserved bottle/container for sampling and transfer to an appropriately preserved container.
2. Remove the cap. Do not place the cap on the ground or elsewhere where it might become contaminated.
3. Carefully dip the container into the water just below the surface (or as directed by project-specific planning documents), and allow the bottle to fill. Sample bottles for volatile analysis must be filled with no headspace. Avoid contacting the bottom of the water body because this will disturb sediment that may interfere with the surface water sample.
4. Retrieve the container and carefully replace the cap securely. If using a container other than the sample bottle, pour the water from that container into the sample bottle and replace the cap securely.
5. Use a clean paper towel to clean and dry the outside of the container.
6. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.
7. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

Constituents measured in grab samples collected near the water surface are only indicative of conditions near the surface of the water and may not be a true representation of the total concentration distributed throughout the water column and in the cross-section. Therefore, as possible based on site conditions, the sampler may be required to augment dip samples with samples that represent both dissolved and suspended constituents and both vertical and horizontal distributions.

CAUTION

In areas prone to natural hazards such as alligators and snakes, etc., always use a buddy as a watch. Always have and use a lifeline or throwable device to extract persons who could potentially fall into the water. Be attentive to the signs, possible mounds indicating nests, and possible slides into the water. Remember that although snakes are typically encountered on the ground, it is not unheard of to see them on low-hanging branches. Be attentive to your surroundings because these may indicate that hazards are nearby.

~~Weighted Bottle Sampling~~

~~A grab sample can also be collected using a weighted holder that allows a bottle to be lowered to any desired depth, opened for filling, closed, and returned to the surface. This allows discrete sampling with depth. Several of these samples can be combined to provide a vertical composite. Alternatively, an open bottle can be lowered to the bottom and raised to the surface at a uniform rate so that the bottle collects sample throughout the total depth and is just filled on reaching the surface. The resulting sample using either method will roughly approach what is known as a depth-integrated sample.~~

~~A closed weighted bottle sampler consists of glass or plastic bottle with a stopper, a weight and/or holding device, and lines to open the stopper and lower or raise the bottle. The general procedure for sampling with this device is as follows:~~

1. ~~Gently lower the sampler to the desired depth so as not to remove the stopper prematurely (watch for bubbles).~~
2. ~~When the desired depth is reached, pull out the stopper with a sharp jerk of the stopper line.~~
3. ~~Allow the bottle to fill completely, as evidenced by the absence of air bubbles.~~
4. ~~Raise the sampler and cap the bottle.~~
5. ~~Use a paper towel to clean and dry the outside of the container. This bottle can be used as the sample container as long as the bottle is an approved container type.~~
6. ~~Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.~~
7. ~~Proceed with the handling and processing of each sample container as described in SOP SA-6.2.~~

~~Kemmerer Sampler~~

~~If samples are desired at a specific depth, and the parameters to be measured do not require a Teflon-coated sampler, a standard Kemmerer sampler may be used. The Kemmerer sampler is a brass, stainless steel or acrylic cylinder with rubber stoppers that leave the ends open while it is lowered in a vertical position (thus allowing free passage of water through the cylinder). A "messenger" is sent down the line when the sampler is at the designated depth to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill sample bottles. The general procedure for sampling with this device is as follows:~~

1. ~~Gently lower the sampler to the desired depth.~~
2. ~~When the desired depth is reached, send down the messenger to close the cylinder and then raise the sampler.~~
3. ~~Open the sampler valve to fill each sample bottle (filling bottles for volatile analysis first).~~
4. ~~Use a paper towel to clean and dry the outside of the container.~~
5. ~~Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.~~
6. ~~Proceed with the handling and processing of each sample container as described in SOP SA-6.2.~~

~~6.1.2 Surface Water Sampling Techniques~~

~~Samples collected during site investigations may be grab samples or composite samples. The following general procedures apply to various types of surface water collection techniques:~~

- ~~If a clean, pre-preserved sample container is not used, rinse the sample container least once with the water to be sampled before the sample is collected. This is not applicable when sample containers are provided pre-preserved because doing so will wash some or all of the preservative out of the bottle.~~
- ~~For sampling moving water, collect the farthest downstream sample first, and continue sample collection in an upstream direction. In general, work from zones suspected of low contamination to zones of high contamination.~~
- ~~Take care to avoid excessive agitation of the water because loss of volatile constituents could result.~~
- ~~When obtaining samples in 40 mL vials with septum-lined lids for volatile organics analysis, fill the container completely (with a meniscus) to exclude any air space in the top of the bottle and to be sure that the Teflon liner of the septum faces in after the vial is filled and capped. Turn the vial upside down and tap gently on your wrist to check for air bubbles. If air bubbles rise in the bottle, add additional sample volume to the container.~~
- ~~Do not sample at the surface, unless sampling specifically for a known constituent that is immiscible and on top of the water. Instead, invert the sample container, lower it to the approximate depth, and hold it at about a 45 degree angle with the mouth of the bottle facing upstream.~~

~~6.2 Onsite Water Quality Testing~~

~~Onsite water quality testing shall be conducted as described in SOP SA-1.1.~~

6.3 Sediment Sampling

6.3.1 General

If composite surface water samples are collected, sediment samples are usually collected at the same locations as the associated surface water samples. If only one sediment sample is to be collected, the sampling location shall be approximately at the center of the water body, in a depositional area if possible based on sample location restraints (see below), unless the SAP states otherwise.

Generally, coarser-grained sediments are deposited near the headwaters of reservoirs. Bed sediments near the center of a water body will be composed of fine-grained materials that may, because of their lower porosity and greater surface area available for adsorption, contain greater concentrations of contaminants. The shape, flow pattern, bathymetry (i.e., depth distribution), and water circulation patterns must all be considered when selecting sediment sampling sites. In streams, areas likely to have sediment accumulation (e.g., bends, behind islands or boulders, quiet shallow areas or very deep, low-velocity areas) shall be sampled, in general, and areas likely to show net erosion (i.e., high-velocity, turbulent areas) and suspension of fine solid materials shall be generally avoided. Follow instructions in the SAP, as applicable.

Chemical constituents associated with bottom material may reflect an integration of chemical and biological processes. Bottom samples reflect the historical input to streams, lakes, and estuaries with

respect to time, application of chemicals, and land use. Bottom sediments (especially fine-grained material) may act as a sink or reservoir for adsorbed heavy metals and organic contaminants (even if water column concentrations are less than detection limits). Therefore, it is important to minimize the loss of low-density "fines" during any sampling process.

Samples collected for volatile organic compound (VOC) analysis must be collected prior to any sample homogenization. Regardless of the method used for collection, the aliquot for VOC analysis must be collected directly from the sampling device (hand auger bucket, scoop, trowel), to the extent practical. If a device such as a dredge is used, the aliquot should be collected after the sample is placed in the mixing container prior to mixing.

In some cases, the sediment may be soft and not lend itself to collection by plunging Encore™ or syringe samplers into the sample matrix. In these cases, it is appropriate to open the sampling device, (Encore™ barrel or syringe) prior to sample collection, and carefully place the sediment in the device, filling it fully with the required volume of sample.

On active or former military sites, ordnance items may be encountered in some work areas. Care should be exercised when handling site media (such as if unloading a dredge as these materials may be scooped up). If suspected ordnance items are encountered, stop work immediately, move to shore and notify the Project Manager and Health and Safety Manager.

All relevant information pertaining to sediment sampling shall be documented as applicably described in SOP SA-6.3 and Attachment B or an equivalent electronic form.

6.3.2 Sampling Equipment and Techniques for Bottom Materials

A bottom-material sample may consist of a single scoop or core, or may be a composite of several individual samples in the cross section. Sediment samples may be obtained using onshore or offshore techniques.

SAFETY REMINDER

The following health and safety provisions apply when working on/over/near water:

- At least two people are required to be present at the sampling location in situations where the water depth and/or movement deem it necessary, each wearing a USCG-approved Personal Flotation Devices
- A minimum of three people are required if *any* of the following conditions are anticipated or observed:
  - Work in a waterway that is turbulent *or* swift that could sweep a sampler down stream should he or she fall in accidentally.
  - The underwater walking surface (e.g., stream/river bed) is suspected or observed to involve conditions that increase the potential for a worker to fall into the water. Examples include large/uneven rocks or boulders, dense mud or sediment that could entrap worker's feet, etc.
  - Waterway is tidal, and conditions such as those listed above could rapidly change.

The third person in the above condition must be equipped and prepared to render emergency support [e.g., lifeline, tethered Personal Flotation Device (Throwable Type IV, life saver), skiff, means to contact external emergency response support, etc.]

The following samplers may be used to collect sediment samples:

- Scoop sampler
- Dredge samplers
- Coring samplers

Each type of sampler is discussed below.

In general, the following equipment if necessary for obtaining sediment samples:

- Required sampling equipment, which may include a scoop sampler, dredge sampler, coring sampler, or stainless steel or pre-cleaned disposable trowel.
- Stainless bowl or pre-cleaned disposable bowl to homogenize sample.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in the project-specific planning document.
- Required PPE as directed in the project-specific planning document, which may include:
  - Nitrile surgeon's or latex gloves (layered as necessary).
  - Safety glasses.
  - Other items identified on the Safe Work Permit that may be required based on location-specific requirements (e.g., hearing protection, steel-toed work boots, hard hat). These provisions will be listed in the HASP or addressed by the FOL and/or SSO.
  - Required paperwork (see SOP SA-6.3 and Attachments A and B to this SOP).
  - Required decontamination equipment.
  - Required sample containers.
  - Sealable polyethylene bags (e.g., Ziploc® baggies).
  - Heavy-duty cooler.
  - Ice.
  - Paper towels and garbage bags.
  - Chain-of-custody records and custody seals.

Scoop Sampler

A scoop sampler consists of a pole to which a jar or scoop is attached. The pole may be made of bamboo, wood, PVC, or aluminum and be either telescoping or of fixed length. The scoop or jar at the end of the pole is usually attached using a clamp.

If the water body can be sampled from the shore or if the sampler can safely wade to the required location, the easiest and best way to collect a sediment sample is to use a scoop sampler. Scoop sampling also reduces the potential for cross-contamination. The general scoop sampling procedure is as follows:

1. Reach over or wade into the water body.
2. While facing upstream (into the current), scoop the sampler along the bottom in an upstream direction. Although it is very difficult not to disturb fine-grained materials at the sediment-water interface when using this method, try to keep disturbances to a minimum.

Dredge Samplers

Dredges are generally used to sample sediments that cannot easily be obtained using coring devices (e.g., coarse-grained or partially cemented materials) or when large quantities of sample are required. Dredges generally consist of a clam shell arrangement of two buckets. The buckets may either close upon impact or be activated by use of a "messenger." Some dredges are heavy and may require use of a winch and crane assembly for sample retrieval. The three major types of dredges are Peterson, Eckman and Ponar.

The Peterson dredge is used when the bottom is rocky, in very deep water, or when the flow velocity is high. The Peterson dredge shall be lowered very slowly as it approaches bottom, because it can force out and miss lighter materials if allowed to drop freely.

The Eckman dredge has only limited usefulness. It performs well where bottom material is unusually soft, as when covered with organic sludge or light mud. It is unsuitable, however, for sandy, rocky, and hard bottoms and is too light for use in streams with high flow velocities.

The Ponar dredge is a Peterson dredge modified by the addition of side plates and a screen on the top of the sample compartment. The screen over the sample compartment permits water to pass through the sampler as it descends, thus reducing the "shock wave." The Ponar dredge is easily operated by one person in the same fashion as the Peterson dredge. The Ponar dredge is one of the most effective samplers for general use on all types of substrates.

The general procedure for using dredge samplers is as follows:

1. Gently lower the dredge to the desired depth.
2. When the desired depth is reached, send the messenger down to cable to close the cylinder and then carefully raise the sampler.
3. Open the sampler to retrieve the sediment.
4. Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis *prior to* homogenization. Homogenize the remainder of the sediment collected.
5. Fill the containers for all analyses other and VOCs.

6. Use a paper towel to clean and dry the outside of each container.

7. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

SAFETY REMINDER

Safety concerns using these dredges include lifting hazards, pinches, and compressions (several pinch points exist within the jaws and levers). In all cases, handle the dredge by the rope to avoid capturing fingers/hands.

Coring Samplers

Coring samplers are used to sample vertical columns of sediment. Many types of coring devices have been developed depending on the depth of water from which the sample is to be obtained, the nature of the bottom material, and the length of core to be collected. They vary from hand-push tubes to electronic vibrational core tube drivers.

Coring devices are particularly useful in pollutant monitoring because turbulence created by descent through the water is minimal, thus the fines at the sediment-water interface are only minimally disturbed. The sample is withdrawn intact, permitting the removal of only those layers of interest.

In shallow, wadeable waters, the use of a core liner or tube manufactured of Teflon or plastic is recommended for the collection of sediment samples. Caution should be exercised not to disturb the bottom sediments when the sample is obtained by wading in shallow water. The general procedure to collecting a sediment sample with a core tube is as follows:

1. Push the tube into the substrate until 4 inches or less of the tube is above the sediment-water interface. When sampling hard or coarse substrates, a gentle rotation of the tube while it is being pushed will facilitate greater penetration and decrease core compaction.

2. Cop the top of the tube to provide suction and reduce the chance of losing the sample.

3. Slowly extract the tube so as not to lose sediment from the bottom of the tube. Cap the bottom of the tube before removing it from the water. This will also help to minimize loss of sample.

4. Transfer the sediment to the bowl in which it will be homogenized. Fill the sample bottle(s) for volatile analysis prior to homogenization. Homogenize the remainder of the sediment collected.

5. Fill the containers for all analyses other and VOCs.

6. Use a paper towel to clean and dry the outside of each container.

7. Affix a sample label to each container, ensuring that each label is completely carefully, clearly, and completely, addressing all of the categories described in SOP SA-6.3.

8. Proceed with the handling and processing of each sample container as described in SOP SA-6.2.

In deeper, non-wadeable water bodies, sediment cores may be collected from a bridge or boat using different coring devices such as Ogeechee Sand Pounders, gravity cores, and vibrating coring devices.

All three devices utilize a core barrel with a core liner tube system. The core liners can be removed from the core barrel and replaced with a clean core liner after each sample. Before extracting the sediment from the coring tubes, the clear supernatant above the sediment-water interface in the core should be decanted from the tube. This is accomplished by turning the core tube to its side and gently pouring the liquid out until fine sediment particles appear in the waste liquid. Post-retrieval processing of samples is the same as above.

7.0 REFERENCES

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Kittrell, F. W., 1969. A Practical Guide to Water Quality Studies of Streams. U.S. Federal Water Pollution Control Administration, Washington, D.C., 135 p.

U.S. EPA, 1984. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-84-017.

U.S. EPA, 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Water Surveillance Branch, USEPA Surveillance and Analytical Division, Athens, Georgia.

U.S. Geological Survey, 1977. National Handbook of Recommended Methods for Water-Data Acquisition. Office of Water Data Coordination, USGS, Reston, Virginia.

ATTACHMENT A
SURFACE WATER SAMPLE LOG SHEET

Tetra Tech NUS, Inc. **SURFACE WATER SAMPLE LOG SHEET**

Page___ of___

Project Site Name: _____
Project No.: _____

[] Stream
[] Spring
[] Pond
[] Lake
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

Date: / Time: / Depth: / Method:	Color Visual	pH Standard	S.C. mS/cm	Temp. Degrees C	Turbidity NTU	DO mg/l	Salinity %	Other NA

Analysis	Preservative	Container Requirements	Collected

Circle if Applicable:		Signature(s):
MS/MSD	Duplicate ID No.:	

ATTACHMENT B
SOIL & SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page ___ of ___

Project Site Name: _____ Sample ID No.: _____
Project No.: _____ Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date:	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time:			
Method:			
Monitor Reading (ppm):			

COMPOSITE SAMPLE DATA:

Date:	Time	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings				
(Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle If Applicable:

MS/MSD

Duplicate ID No.:

Signature(s):

APPENDIX C
GUIDANCE ON SAMPLING DESIGN AND SAMPLE COLLECTION

C.1 Defining the Sampling Program

Many factors are considered in developing a sampling program for surface water and/or sediment, including study objectives, accessibility, site topography, physical characteristics of the water body (e.g., flow and mixing), point and diffuse sources of contamination, and personnel and equipment available to conduct the study. For waterborne constituents, dispersion depends on vertical and lateral mixing within the body of water. For sediment, dispersion depends on bottom current or flow characteristics, sediment characteristics (e.g., density, size), and geochemical properties (that affect adsorption/desorption). The hydrogeologist developing the sampling plan must therefore know not only the mixing characteristics of streams and lakes but must also understand the role of fluvial-sediment transport, deposition, and chemical sorption.

C.1.1 Sampling Program Objectives

The scope of the sampling program must consider the sources and potential pathways for transport of contamination to or within a surface water body. Sources may include point sources (leaky tanks, outfalls, etc.) or nonpoint sources (e.g., contaminated runoff). The major pathways for surface water contamination (not including airborne deposition) are overland runoff, leachate influx to the water body, direct waste disposal (solid or liquid) into the water body, and groundwater flow influx from upgradient. The relative importance of these pathways, and therefore the design of the sampling program, is controlled by the physiographic and hydrologic features of the site, the drainage basin(s) that encompasses the site, and the history of site activities.

Physiographic and hydrologic features to be considered include slopes and runoff direction, areas of temporary flooding or pooling, tidal effects, artificial surface runoff controls such as berms or drainage ditches (and when they were constructed relative to site operation), and locations of springs, seeps, marshes, etc. In addition, the obvious considerations such as the locations of man-made discharge points to the nearest stream (intermittent or flowing), pond, lake, estuary, etc. shall be considered.

A more subtle consideration in designing the sampling program is the potential for dispersion of dissolved or sediment-associated contaminants away from the source. The dispersion could lead to a more homogeneous distribution of contamination at low or possibly non-detectable concentrations. Such dispersion does not, however, always readily occur. For example, obtaining a representative sample of contamination from a main stream immediately below an outfall or a tributary is difficult because the inflow frequently follows a stream bank with little lateral mixing for some distance. Sampling alternatives to overcome this situation include: (1) moving the sampling location far enough downstream to allow for adequate mixing, or (2) collecting integrated samples in a cross section. Also, non-homogeneous distribution is a particular problem with regard to sediment-associated contaminants, which may accumulate in low-energy environments (coves, river bends, deep spots, or even behind boulders) near or distant from the source while higher-energy areas (main stream channels) near the source may show no contaminant accumulation.

The distribution of particulates within a sample itself is an important consideration. Many organic compounds are only slightly water soluble and tend to adsorb onto particulate matter. Nitrogen, phosphorus, and heavy metals may also be transported by particulates. Samples must be collected with a representative amount of suspended material; transfer from the sampling device shall include transferring a proportionate amount of the suspended material.

C.1.2 Location of Sampling Stations

Accessibility is the primary factor affecting sampling costs. The desirability and utility of a sample for analysis and consideration of site conditions must be balanced against the costs of collection as controlled by accessibility. Bridges or piers are the first choice for locating a sampling station on a stream because bridges provide ready access and also permit the sampling technician to sample any point across the stream. A boat or pontoon (with an associated increase in cost) may be needed to sample locations on lakes, reservoirs, or larger rivers. Frequently, however, a boat will take longer to cross a water body and will hinder manipulation of the sampling equipment. Wading for samples is not recommended unless it is known that contaminant levels are low so that skin contact will not produce adverse health effects. This provides a built in margin of safety in the event that wading boots or other protective equipment should fail to function properly. If it is necessary to wade into the water body to obtain a sample, the sampler shall be careful to minimize disturbance of bottom sediments and must enter the water body downstream of the sampling location. If necessary, the sampling technician shall wait for the sediments to settle before taking a sample.

Under ideal and uniform contaminant dispersion conditions in a flowing stream, the same concentrations of each contaminant would occur at all points along the cross section. This situation is most likely downstream of areas of high turbulence. Careful site selection is needed to ensure, as nearly as possible, that samples are taken where uniform flow or deposition and good mixing conditions exist.

The availability of stream flow and sediment discharge records can be an important consideration in choosing sampling sites in streams. Stream flow data in association with contaminant concentration data are essential for estimating the total contaminant loads carried by the stream. If a gaging station is not conveniently located on a selected stream, the project hydrogeologist shall explore the possibility of obtaining stream flow data by direct or indirect methods. Remember these locations are also where you may encounter natural hazards as these are areas where they hunt. Always exercise extreme caution.

C.1.3 Frequency of Sampling

The sampling frequency and objectives of the sampling event will be defined by the project planning documents. For single-event site or area characterization sampling, both bottom material and overlying water samples shall be collected at the specified sampling stations. If valid data are available on the distribution of a contaminant between the solid and aqueous phases, it may be appropriate to sample only one phase, although this is not often recommended. If samples are collected primarily for monitoring purposes (i.e., consisting of repetitive, continuing measurements to define variations and trends at a given location), water samples should be collected at a pre-established and constant interval as specified in the project plans (often monthly or quarterly and during droughts and floods). Samples of bottom material should generally be collected from fresh deposits at least yearly, and preferably seasonally, during both spring and fall.

The variability in available water quality data shall be evaluated before determining the number and collection frequency of samples required to maintain an effective monitoring program.

~~C.2 Surface Water Sample Collection~~

~~C.2.1 Streams, Rivers, Outfalls and Drainage Features~~

~~Methods for sampling streams, rivers, outfalls, and drainage features (ditches, culverts) at a single point vary from the simplest of hand sampling procedures to the more sophisticated multi-point sampling techniques known as the equal width increment (EWI) method or the equal discharge increment (EDI) methods (see below).~~

Samples from different depths or cross-sectional locations in the watercourse taken during the same sampling episode shall be composited. However, samples collected along the length of the watercourse or at different times may reflect differing inputs or dilutions and therefore shall not be composited. Generally, the number and type of samples to be taken depend on the river's width, depth, and discharge and on the suspended sediment the stream or river transports. The greater the number of individual points that are sampled, the more likely that the composite sample will truly represent the overall characteristics of the water.

In small streams less than about 20 feet wide, a sampling site can generally be found where the water is well mixed. In such cases, a single grab sample taken at mid-depth in the center of the channel is adequate to represent the entire cross section.

For larger streams, at least one vertical composite shall be taken with one sample each from just below the surface, at mid-depth, and just above the bottom. The measurement of dissolved oxygen (DO), pH, temperature, conductivity, etc., shall be made on each aliquot of the vertical composite and on the composite itself. For rivers, several vertical composites shall be collected, as directed in the project planning documents.

C.2.2 Lakes, Ponds and Reservoirs

Lakes, ponds, and reservoirs have a much greater tendency to stratify than rivers and streams. The relative lack of mixing requires that more samples be obtained. The number of water sampling sites on a lake, pond, or impoundment will vary with the size and shape of the basin. In ponds and small lakes, a single vertical composite at the deepest point may be sufficient. Similarly, measurement of DO, pH, temperature, etc. is to be conducted on each aliquot of the vertical composite and on the composite itself. In naturally formed ponds, the deepest point may have to be determined empirically; in impoundments, the deepest point is usually near the dam.

In lakes and larger reservoirs, several vertical composites shall be composited to form a single sample if a sample representative of the water column is required. These vertical composites are often collected along a transect or grid. In some cases, it may be of interest to form separate composites of epilimnetic and hypolimnetic zones. In a stratified lake, the epilimnion is the thermocline that is exposed to the atmosphere. The hypolimnion is the lower, "confined" layer that is only mixed with the epilimnion and vented to the atmosphere during seasonal "overturn" (when density stratification disappears). These two zones may thus have very different concentrations of contaminants if input is only to one zone, if the contaminants are volatile (and therefore vented from the epilimnion but not the hypolimnion), or if the epilimnion only is involved in short-term flushing (i.e., inflow from or outflow to shallow streams). Normally, however, a composite consists of several vertical composites with samples collected at various depths.

In lakes with irregular shape and with bays and coves that are protected from the wind, separate composite samples may be needed to adequately represent water quality because it is likely that only poor mixing will occur. Similarly, additional samples are recommended where discharges, tributaries, land use characteristics, and other such factors are suspected of influencing water quality.

Many lake measurements are now made in situ using sensors and automatic readout or recording devices. Single and multi-parameter instruments are available for measuring temperature, depth, pH, oxidation-reduction potential (ORP), specific conductance, DO, some cations and anions, and light penetration.

C.2.3 ~~Estuaries~~

~~Estuarine areas are, by definition, zones where inland freshwaters (both surface and ground) mix with oceanic saline waters. Knowledge of the estuary type may be necessary to determine sampling locations. Estuaries are generally categorized into one of the following three types dependent on freshwater inflow and mixing properties:~~

- ~~Mixed Estuary - characterized by the absence of a vertical halocline (gradual or no marked increase in salinity in the water column) and a gradual increase in salinity seaward. Typically, this type of estuary is shallow and is found in major freshwater sheet flow areas. Because this type of estuary is well mixed, sampling locations are not critical.~~

- ~~Salt Wedge Estuary - characterized by a sharp vertical increase in salinity and stratified freshwater flow along the surface. In these estuaries, the vertical mixing forces cannot override the density differential between fresh and saline waters. In effect, a salt wedge tapering inland moves horizontally back and forth with the tidal phase. If contamination is being introduced into the estuary from upstream, water sampling from the salt wedge may miss it entirely.~~

- ~~Oceanic Estuary - characterized by salinities approaching full-strength oceanic waters. Seasonally, freshwater inflow is small, with the preponderance of the fresh saline water mixing occurring near or at the shore line.~~

~~Sampling in estuarine areas is normally based on the tidal phase, with samples collected on successive slack tides (i.e., when the tide turns). Estuarine sampling programs shall include vertical salinity measurements at 1- to 5-foot increments, coupled with vertical DO and temperature profiles.~~

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		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject SOIL SAMPLING		Approved Tom Johnston T E Johnston	

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Thin-Walled Tube Sampler - A thin-walled metal tube (also called a Shelby tube) used to recover relatively undisturbed soil samples. These tubes are available in various sizes, ranging from 2 to 5 inches outside diameter (OD) and from 18 to 54 inches in length.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, selecting proposed sampling locations, and selecting field procedures used in the collection of soil samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager establishes the need for test pits or trenches and determines their approximate locations and dimensions.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan. This will include (but not be limited to) performing air quality monitoring during sampling, boring, and excavation activities and to ensure that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO/designee may also be required to advise the FOL on other safety-related matters regarding boring, excavation, and sampling, such as mitigative measures to address potential hazards from unstable trench walls, puncturing of drums or other hazardous objects, etc.

Field Operations Leader (FOL) - This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is responsible for finalizing the locations for collection of surface, near-surface, and subsurface (hand and machine borings, test pits/trenches) soil samples. He/she is ultimately responsible for the sampling and backfilling of boreholes, test pits, and trenches and for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP and/or other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, boring logs, test pit logs, container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

Competent Person - A Competent Person, as defined in 29 CFR 1929.650 of Subpart P - Excavations, means one who is capable of identifying existing and predictable hazards in the surroundings, or working conditions that are unsanitary, hazardous, or dangerous to employees, and who has authorization to take prompt corrective measures to eliminate them.

General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.

- Whenever practical, wear cut-resistant gloves (e.g., leather or heavy cotton work gloves) at least on the hand not using the knife.
- Keep cutting surfaces clean and smooth.
- Secure items to be cut – do not hold them against the opposing hand, a leg, or other body part.
- When transporting glassware, keep it in a hard-sided container such as a cooler so that if there is a fall, you will be less likely to get cut by broken glass.
- DO NOT throw broken sample jars or glass ampoules into garbage bags. Place broken glass and glass ampoules in hard-sided containers such as a cardboard box or directly into a dumpster. DO NOT reach into garbage bags to retrieve any item accidentally thrown away. Empty the contents onto a flat surface to avoid punctures and lacerations from reaching where you cannot see.

Vehicular and Foot Traffic Hazards – When sampling along the roadway or near traffic patterns, follow the following precautions:

- Motorists may be distracted by onsite activities – ASSUME THEY DO NOT SEE YOU OR MEMBERS OF YOUR FIELD CREW.
- DO NOT place obstructions (such as vehicles) along the sides of the road that may cause site personnel to move into the flow of traffic to avoid your activities or equipment or that will create a blind spot.
- **Provide a required free space of travel.** Maintain at least 6 feet of space between you and moving traffic. Where this is not possible, use flaggers and/or signs to warn oncoming traffic of activities near or within the travel lanes.
- Face Traffic. Whenever feasible, if you must move within the 6 feet of the required free space or into traffic, attempt to face moving traffic at all times. Always leave yourself an escape route.
- Wear high-visibility vests to increase visual recognition by motorists.
- Do not rely on the vehicle operator's visibility, judgment, or ability. Make eye contact with the driver. Carefully and deliberately use hand signals so they will not startle or confuse motorists or be mistaken for a flagger's direction before moving into traffic.
- Your movements may startle a motorist and cause an accident, so move deliberately. Do not make sudden movements that might confuse a motorist.

6.0 PROCEDURES

The following procedures address surface and subsurface sampling.

> ***CAUTION***
>
> Each situation must be evaluated individually to determine the applicability and necessity for obtaining a utility clearance ticket/dig permit. Common sense dictates, prior to digging or boring with power equipment, no matter what the depth, or digging by hand in a manner that could damage unprotected underground utilities, that a dig permit is required. See SOP HS-1.0, Utility Locating and Excavation Clearance, for additional clarification. If you do not know or are unsure as to whether a ticket is necessary – **Get the Ticket**.

obtained using a reusable sampling handle (T-handle) that can be provided with the EnCore™ sampler when requested and purchased. Collect the sample in the following manner for each EnCore™ sampler:

1. Scene Safety - Evaluate the area where sampling will occur. Ensure that the area is safe from physical, chemical, and natural hazards. Clear or barricade those hazards that have been identified.

2. Wear the appropriate personal protective equipment (PPE). This will include, at a minimum, safety glasses and nitrile surgeon's gloves. If you must kneel on the ground or place equipment on the surface being sampled, cover the ground surface with plastic to minimize surface contamination of your equipment and clothing. Wear knee pads to protect your knees from kneeling on hard or uneven surfaces.

3. Load the Encore™ sampler into the T-handle with the plunger fully depressed.

4. Expose the area to be sampled using a hand trowel or similar device to remove surface debris.

5. Press the T-handle against the freshly exposed soil surface, forcing soil into the sampler. The plunger will be forced upward as the cavity fills with soil.

6. When the sampler is full, rotate the plunger and lock it into place. If the plunger does not lock, the sampler is not full. This method ensures there is no headspace. Soft soil may require several plunges or forcing soil against a hard surface such as a sample trowel to ensure that headspace is eliminated.

7. Use a paper towel to remove soil from the side of the sampler so a tight seal can be made between the sample cap and the rubber O-ring.

8. With soil slightly piled above the rim of the sampler, force the cap on until the catches hook the side of the sampler.

9. Remove any surface soil from the outside of the sampler and place in the foil bag provided with the sampler. Good work hygiene practices and diligent decontamination procedures prevents the spread of contamination even on the outside of the containers.

10. Label the bag with appropriate information in accordance with SOP SA-6.3.

11. Place the full sampler inside a lined cooler with ice and cool to 4°C ± 2 °C. Make sure any required trip blanks and temperature blanks are also in the cooler. Secure custody of the cooler in accordance with SOP SA-6.3.

12. Typically, collect three Encore™ samplers at each location. Consult the SAP or laboratory to determine the required number of Encore™ samplers to be collected.

13. The T-handle shall be decontaminated before moving to the next interval or location using a soap and water wash and rinse, and where applicable, the selected solvent as defined in the project planning documents.

Using this type of sampling device eliminates the need for field preservation and the shipping restrictions associated with preservatives. A complete set of instructions is included with each Encore™ sampler.

After the Encore™samples are collected, they should be placed on ice immediately and delivered to the laboratory within 48 hours (following the chain-of-custody and documentation procedures outlined in SOP SA-6.1). Samples must be preserved by the laboratory within 48 hours of sample collection.

5. Record the sample weight to the nearest 0.01 gram in the field logbook and/or on the sample log sheet.

6. Extrude the weighed soil sample into the methanol-preserved sample bottle taking care not to contact the sample container with the syringe.

7. If dirty, wipe soil particles from the threads of the bottle and cap. Cap the bottle tightly.

8. After capping the bottle, swirl the sample (do not shake) in the methanol and break up the soil such that all of the soil is covered with methanol.

9. Place the sample on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

Sodium Bisulfate Preservation (Low Level):

> ***CAUTION***
> Care should be taken when adding the soil to the sodium bisulfate solution. A chemical reaction of soil containing carbonates (limestone) may cause the sample to effervesce or the vial to possibly explode. To avoid this hazard or hazards of this type, a small sample aliquot should be subjected to the sample preservative. If it effervesces in an open air environment, utilize an alternative method such as Encore™ or 2-ounce jar.

Bottles may be prepared in the laboratory or in the field with sodium bisulfate solution. Samples to be preserved in the field using the sodium bisulfate method are to be prepared and collected as follows:

1. Add 1 gram of sodium bisulfate to 5 mL of laboratory-grade deionized water in a 40 to 60 mL glass vial with septum-lined lid.

2. Collect the soil sample and record the sample weight to the nearest 0.01 gram in the field logbook or on the sample log sheet as described for methanol preservation

3. Add the weighed sample to the sample vial.

4. Collect duplicate samples using the methanol preservation method on a one-for-one sample basis because it is necessary for the laboratory to perform both low-level and medium-level analyses.

5. Place the samples on ice immediately and prepare for shipment to the laboratory as described in SOP SA-6.1.

> ***NOTE***
> If lower detection limits are necessary, an option to field preserving with sodium bisulfate may be to collect EnCore™ samplers at a given sample location. Consult the planning documents to determine whether this is required. If it is, collect samples in accordance with the Encore™ sampling procedure above and then send all samplers to the laboratory to perform the required preservation and analyses.

6.2.2 Procedure for Collecting Soil Samples for Non-Volatile Analyses

Samples collected for non-volatile analyses may be collected as either grab or composite samples as follows:

REMEMBER

If you are digging near a marked utility (within the diameter of an underground utility that has been marked plus 18 inches), you must first locate the utility through vacuum extraction or hand digging to ensure that your activities will not damage the utility.

- Complete an Equipment Inspection Checklist for the drill rig or direct-push technology (DPT) rig. This checklist will be provided in the HASP.
- Review the Safe Work Permit prior to conducting the activity.
- Review the activity to be conducted.

2. Remove all surface debris (e.g., vegetation, roots, twigs, etc.) from the specific sampling location and drill and/or clean out the borehole to the desired sampling depth. Be careful to minimize potential disturbance of the material to be sampled. In saturated material, withdraw the drill bit slowly to prevent loosening of the soil around the borehole and to maintain the water level in the hole at or above groundwater level.

CAUTION

The use of bottom-discharge bits or jetting through an open-tube sampler to clean out the borehole shall not be allowed. Only the use of side-discharge bits is permitted.

3. Determine whether a stationary piston-type sampler is required to limit sample disturbance and aid in retaining the sample. Either the hydraulically operated or control rod activated-type of stationary piston sampler may be used.
4. Prior to inserting the tube sampler into the borehole, check to ensure that the sampler head contains a check valve. The check valve is necessary to keep water in the rods from pushing the sample out the tube sampler during sample withdrawal. In addition, the check valve maintains a positive suction within the tube to help retain the sample.
5. A stainless steel tube sampler is typically used to minimize chemical reaction between the sample and the sampling tube.
6. With the sampling tube resting on the bottom of the hole and the water level in the boring at groundwater level or above, push the tube into the soil with a continuous and rapid motion, without impacting or twisting. If the soil is too hard to penetrate by pushing alone, careful hammering may be used by minimizing drop distance (tapping) of the hammer. Before pulling the tube, turn it at least one revolution to shear the sample off at the bottom. In no case shall the tube be pushed farther than the length provided for the soil sample. Allow about 3 inches in the tube for cuttings and sludge.
7. Upon removal of the sampling tube from the hole, measure the length of sample in the tube and also the length penetrated.
8. Remove disturbed material in the upper end of the tube and measure the length of sample again.
9. After removing at least 1 inch of soil from the lower end, place enough packing material (clean inert material such as paper or cloth) tightly in each end of the Shelby tube and then pour melted wax into each end to make at least a ½-inch wax plug and then add more packing material to fill the voids at both ends.

6.3 Surface Soil Sampling

The simplest, most direct method of collecting surface soil samples for subsequent analysis is by use of a stainless steel shovel, hand auger, soil corer, or stainless steel or disposable plastic trowel.

> ***NOTE***
> Multiple depth intervals are used to describe surface soil. Sometimes surface soil is defined as soil from 0 to 2 inches below ground surface (bgs), and sometimes it is defined as soil from other depths such as 0 to 2 feet bgs. Ensure that the definition of surface soil depth is clear before collecting surface soil samples.
>
> For the purposes of instruction, the terms "surface soil" and "near-surface soil" are used in this SOP as follows:
>
> - Surface soil - 0 to 6 inches bgs
> - Near-surface soil - 6 to 18 inches bgs
>
> If these intervals are defined differently in the planning documents, substitute the appropriate depth ranges.

In general, the following equipment is necessary for obtaining surface soil samples:

- Stainless steel or pre-cleaned disposable trowel.
- Stainless steel hand auger, soil corer, or shovel.
- Real-time air monitoring instrument (e.g., PID, FID) as directed in project planning document.
- Required PPE.
  - Nitrile surgeon's or latex gloves may be used, layered as necessary.
  - Safety glasses
  - Other – Items identified on the Safe Work Permit may be required based on location-specific requirements such as hearing protection, steel-toed work boots, and a hard hat when working near a drill rig. These provisions will be listed in the HASP or directed by the FOL and/or SSO.

> ***Safety Reminder***
> The use of latex products may elicit an allergic reaction in some people. Should this occur, remove the latex gloves, treat for an allergic reaction, and seek medical attention as necessary.

- Required paperwork (see SOP SA-6.3 and Attachment A of this SOP)
- Required decontamination equipment
- Required sample container(s)
- Wooden stakes or pin flags

3. Follow steps 1 through 9 of Section 6.3.

6.5 Subsurface Soil Sampling With a Hand Auger

A hand augering system generally consists of a variety of stainless steel bucket bits (approximately 6.5 inches long and 2, 2.75, 3.25, and 4 inches in diameter), series of extension rods (available in 2-, 3-, 4- and 5-inch lengths), and a T-handle connected to extension rods and to the auger bucket. A larger-diameter bucket bit is commonly used to bore a hole to the desired sampling depth and then it is withdrawn. The larger-diameter bit is then replaced with a smaller-diameter bit, lowered down the hole, and slowly turned into the soil to the completion depth (approximately 6 inches). The apparatus is then withdrawn and the soil sample collected.

The hand auger can be used in a wide variety of soil conditions. It can be used to sample soil either from the surface, or to depths in excess of 12 feet. However, the presence of subsurface rocks and landfill material and collapse of the borehole normally limit sampling depth.

To accomplish soil sampling using a hand augering system, the following equipment is required:

- Complete hand auger assembly (variety of bucket bit sizes)
- Stainless steel mixing bowls
- The equipment listed in Section 6.3
- Miscellaneous hand tools as required to assemble and disassemble the hand auger units

CAUTION

Potential hazards associated with hand augering include:

- Muscle strain and sprain due to over twisting and/or over compromising yourself.
- Equipment failure due to excessive stress on the T-handle or rods through twisting. Failure of any of these components will result in a sudden release and potential injury due to that failure.

As in all situations, any intrusive activities that could damage underground utilities shall be proceeded by a Dig/Excavation permit/ticket. Call the Utility Locating service in the area or your Project Health and Safety Officer for more information. When in doubt – **Get the Ticket!**

To obtain soil samples using a hand auger, use the following procedure:

1. Wearing designated PPE, attach a properly decontaminated bucket bit to a clean extension rod and attach the T-handle to the extension rod.
2. Clear the area to be sampled of any surface debris (vegetation, twigs, rocks, litter, etc.).
3. Twist the bucket into the ground while pushing vertically downward on the auger. The cutting shoes fill the bucket as it is advanced into the ground.
4. As the auger bucket fills with soil, periodically remove any unneeded soil.

- Job rotation – Share the duties so that repetitive actions do not result in fatigue and injury.
- Increase break frequencies as needed, especially as ambient conditions of heat and/or cold stress may dictate.
- Do not force the hand tools or use cheater pipes or similar devices to bypass an obstruction. Move to another location near the sampling point. Exerting additional forces on the sampling devices can result in damage and/or failure that could potentially injure someone in the immediate vicinity.
- Do not over compromise yourself when applying force to the soil corer or hand auger. If there is a sudden release, it could result in a fall or muscle injury due to strain.

6.6 Subsurface Soil Sampling with a Split-Barrel Sampler

A split-barrel (split-spoon) sampler consists of a heavy carbon steel or stainless steel sampling tube that can be split into two equal halves to reveal the soil sample (see Attachment B). A drive head is attached to the upper end of the tube and serves as a point of attachment for the drill rod. A removable tapered nosepiece/drive shoe attaches to the lower end of the tube and facilitates cutting. A basket-like sample retainer can be fitted to the lower end of the split tube to hold loose, dry soil samples in the tube when the sampler is removed from the drill hole. This split-barrel sampler is made to be attached to a drill rod and forced into the ground by means of a 140-pound or larger casing driver.

Safety Reminder

It is intended through the Equipment Inspection for Drill Rigs form provided in the HASP that the hammer and hemp rope, where applicable, associated with this activity will be inspected (no physical damage is obvious), properly attached to the hammer (suitable knots or sufficient mechanical devices), and is in overall good condition.

Split-barrel samplers are used to collect soil samples from a wide variety of soil types and from depths greater than those attainable with other soil sampling equipment.

The following equipment is used for obtaining split-barrel samples:

- Drilling equipment (provided by subcontractor).
- Split-barrel samplers (2-inch OD, 1-3/8-inch ID, either 20 inches or 26 inches long); Larger OD samplers are available if a larger volume of sample is needed.
- Drive weight assembly, 140-pound weight, driving head, and guide permitting free fall of 30 inches.
- Stainless steel mixing bowls.
- Equipment listed in Section 6.3.

The following steps shall be followed to obtain split-barrel samples (Steps 1 through 4 are typically performed by the drilling subcontractor):

1. Attach the split-barrel sampler to the sampling rods.

6.8 ~~Excavation and Sampling of Test Pits and Trenches~~

6.8.1 ~~Applicability~~

~~This subsection presents routine test pit or trench excavation techniques and specialized techniques that are applicable under certain conditions.~~

CAUTION

During the excavation of trenches or pits at hazardous waste sites, several health and safety concerns arise from the method of excavation. No personnel shall enter any test pit or excavation over 4 feet deep except as a last resort, and then only under direct supervision of a Competent Person (as defined in 29 CFR 1929.650 of Subpart P - Excavations). Whenever possible, all required chemical and lithological samples should be collected using the excavator bucket or other remote sampling apparatus. If entrance is required, all test pits or excavations must be stabilized by bracing the pit sides using specifically designed wooden, steel, or aluminum support structures or through sloping and benching. Personnel entering the excavation may be exposed to toxic or explosive gases and oxygen-deficient environments; therefore, monitoring will be conducted by the Competent Person to determine if it is safe to enter. Any entry into a trench greater than 4 feet deep will constitute a Confined Space Entry and must be conducted in conformance with OSHA standard 29 CFR 1910.146. In all cases involving entry, substantial air monitoring, before entry, appropriate respiratory gear and protective clothing determination, and rescue provisions are mandatory. There must be at least three people present at the immediate site before entry by one of the field team members. This minimum number of people will increase based on the potential hazards or complexity of the work to be performed. The reader shall refer to OSHA regulations 29 CFR 1926.650, 29 CFR 1910.120, 29 CFR 1910.134, and 29 CFR 1910.146. High-hazard entries such as this will be supported by members of the Health Sciences Group professionally trained in these activities.

~~Excavations are generally not practical where a depth of more than about 15 to 20 feet is desired, and they are usually limited to a few feet below the water table. In some cases, a pumping system may be required to control water levels within the pit, providing that pumped water can be adequately stored or disposed. If soil data at depths greater than 15 feet are required, the data are usually obtained through test borings instead of test pits.~~

~~In addition, hazardous wastes may be brought to the surface by excavation equipment. This material, whether removed from the site or returned to the subsurface, must be properly handled according to any and all applicable federal, state, and local regulations!~~

6.8.2 ~~Test Pit and Trench Excavation~~

~~Test pits or trench excavations are constructed with the intent that they will provide an open view of subsurface lithology and/or disposal conditions that a boring will not provide. These procedures describe the methods for excavating and logging test pits and trenches installed to determine subsurface soil and rock conditions. Test pit operations shall be logged and documented (see Attachment C).~~

~~Test pits and trenches may be excavated by hand or power equipment to permit detailed descriptions of the nature and contamination of the in-situ materials. The size of the excavation will depend primarily on the following:~~

- ~~The purpose and extent of the exploration~~

~~samples of leachate, groundwater, or sidewall soil can be collected with telescoping poles or similar equipment.~~

~~Dewatering and watering may be required to ensure the stability of the side walls, to prevent the bottom of the pit from heaving, and to keep the excavation stable. This is an important consideration for excavations in cohesionless material below the groundwater table and for excavations left open greater than a day. Liquids removed as a result of dewatering operations must be handled as potentially contaminated materials. Procedures for the collection and disposal of such materials should be discussed in the site-specific project plans.~~

~~Where possible excavations and test pits shall be opened and closed within the same working day. Where this is not possible, the following engineering controls shall be put in place to control access:~~

- ~~Trench covers/street plates~~
- ~~Fences encompassing the entire excavation intended to control access~~
- ~~Warning signs warning personnel of the hazards~~
- ~~Amber flashing lights to demarcate boundaries of the excavation at night.~~

~~Excavations left open will have emergency means to exit should someone accidentally enter.~~

6.8.3 ~~Sampling In Test Pits and Trenches~~

6.8.3.1 ~~General~~

~~Log test pits and trenches as they are excavated in accordance with the Test Pit Log presented in Attachment C. These records include plan and profile sketches of the test pit/trench showing materials encountered, their depth and distribution in the pit/trench, and sample locations. These records also include safety and sample screening information.~~

~~Entry of test pits by personnel is extremely dangerous, shall be avoided unless absolutely necessary, and can occur only after all applicable health and safety and OSHA requirements have been met as stated above. These provisions will be reiterated as appropriate in the project specific HASP.~~

~~The final depth and type of samples obtained from each test pit will be determined at the time the test pit is excavated. Sufficient samples are usually obtained and analyzed to quantify contaminant distribution as a function of depth for each test pit. Additional samples of each waste phase and any fluids encountered in each test pit may also be collected.~~

~~In some cases, samples of soil may be extracted from the test pit for reasons other than waste sampling and chemical analysis, for instance, to obtain geotechnical information. Such information includes soil types, stratigraphy, strength, etc., and could therefore entail the collection of disturbed (grab or bulk) or relatively undisturbed (hand-carved or pushed/driven) samples that can be tested for geotechnical properties. The purposes of such explorations are very similar to those of shallow exploratory or test borings, but often test pits offer a faster, more cost effective method of sampling than installing borings.~~

6.8.3.2 ~~Sampling Equipment~~

~~The following equipment is needed for obtaining samples for chemical or geotechnical analysis from test pits and trenches:~~

- ~~Samples of the test pit material are to be obtained either directly from the backhoe bucket or from the material after it has been deposited on the ground, as follows:~~

  a. ~~The sampler or FOL shall direct the backhoe operator to remove material from the selected depth or location within the test pit/trench.~~

  b. ~~The backhoe operator shall bring the bucket over to a designated location on the sidewall a sufficient distance from the pit (at least 5 feet) to allow the sampler to work around the bucket.~~

  c. ~~After the bucket has been set on the ground, the backhoe operator shall either disengage the controls or shut the machine down.~~

  d. ~~When signaled by the operator that it is safe to do, the sampler will approach the bucket.~~

  e. ~~The soil shall be monitored with a photoionization or flame ionization detector (PID or FID) as directed in the project specific planning documents.~~

  f. ~~The sampler shall collect the sample from the center of the bucket or pile in accordance with surface soil sampling procedures of Section 6.3 or 6.4, as applicable. Collecting samples from the center of a pile or bucket eliminates cross contamination from the bucket or other depth intervals.~~

- ~~If a composite sample is desired, several depths or locations within the pit/trench will be selected, and the bucket will be filled from each area. It is preferable to send individual sample bottles filled from each bucket to the laboratory for compositing under the more controlled laboratory conditions. However, if compositing in the field is required, each sample container shall be filled from materials that have been transferred into a mixing bucket and homogenized. Note that homogenization/compositing is not applicable for samples to be subjected to volatile organic analysis.~~

> ***CAUTION***
>
> Care must be exercised when using the remote sampler described in the next step because of potential instability of trench walls. In situations where someone must move closer than 2 feet to the excavation edge, a board or platform should be used to displace the sampler's weight to minimize the chance of collapse of the excavation edge. Fall protection should also be employed when working near the edges or trenches greater than 6 feet deep. An immediate means to extract people who have fallen into the trench will be immediately available. These means may include ladders or rope anchor points.

- ~~Using the remote sampler shown in Attachment D, samples can be taken at the desired depth from the sidewall or bottom of the pit as follows:~~

  a. ~~Scrape the face of the pit/trench using a long handled shovel or hoe to remove the smeared zone that has contacted the backhoe bucket.~~

  b. ~~Collect the sample directly into the sample jar, by scraping with the jar edge, eliminating the need for sample handling equipment and minimizing the likelihood of cross contamination.~~

  c. ~~Cap the sample jar, remove it from the remote sampler assembly, and package the sample for shipment in accordance with SOP SA-6.3.~~

- ~~Complete documentation as described in SOP SA-6.3 and Attachment C of this SOP.~~

~~Disturbed grab or bulk geotechnical soil samples may be collected for most soil in the same manner as comparable soil samples for chemical analysis. These collected samples may be stored in jars or plastic lined sacks (larger samples), which will preserve their moisture content. Smaller samples of this type are usually tested for their index properties to aid in soil identification and classification; larger bulk samples are usually required to perform compaction tests.~~

~~Relatively undisturbed samples are usually extracted in cohesive soil using thin-walled tube samplers, and such samples are then tested in a geotechnical laboratory for their strength, permeability, and/or compressibility. The techniques for extracting and preserving such samples are similar to those used in performing Shelby tube sampling in borings, except that the sampler is advanced by hand or backhoe, rather than by a drill rig. Also, the sampler may be extracted from the test pit by excavation around the tube when it is difficult to pull it out of the ground. If this excavation requires entry of the test pit, the requirements described in Section 6.8.3.4 shall be followed. The thin walled tube sampler shall be pushed or driven vertically into the floor or steps excavated in the test pit at the desired sampling elevations. Extracting tube samples horizontally from the walls of the test pit is not appropriate because the sample will not have the correct orientation.~~

~~A sledge hammer or backhoe may be used to drive or push the tube into the ground. Place a piece of wood over the top of the sampler or sampling tube to prevent damage during driving/pushing of the sample. Pushing the sampler with a constant thrust is always preferable to driving it with repeated blows, thus minimizing disturbance to the sample. When using a sledge hammer, it is recommended that the sampler be stabilized using a rope/strap wrench or pipe wrench to remove the person's hands holding the sampler from the strike zone. If the sample cannot be extracted by rotating it at least two revolutions (to shear off the sample at the bottom), hook the sampler to the excavator or backhoe and extract. This means an alternative head will be used as a connection point or that multiple choke hitches will be applied to extract the sampler. If this fails and the excavator can dig deeper without potentially impacting subsurface utilities, excavate the sampler. If this fails or if the excavator cannot be used due to subsurface utilities, hand excavate to remove the soil from around the sides of the sampler. If hand excavation requires entry into the test pit, the requirements in Section 6.8.3.4 must be followed. Prepare the sample as described in Steps 9 through 13 in Section 6.2.3, and label, pack and transport the sample in the required manner, as described in SOPs SA-6.3 and SA-6.1.~~

~~6.8.4 Backfilling of Trenches and Test Pits~~

~~All test pits and excavations must be either backfilled, covered, or otherwise protected at the end of each day. No excavations shall remain open during non-working hours unless adequately covered or otherwise protected.~~

~~Before backfilling, the onsite crew may photograph, if required by the project specific work plan, all significant features exposed by the test pit and trench and shall include in the photograph a scale to show dimensions. Photographs of test pits shall be marked to include site number, test pit number, depth, description of feature, and date of photograph. In addition, a geologic description of each photograph shall be entered in the site logbook. All photographs shall be indexed and maintained as part of the project file for future reference.~~

~~After inspection, backfill material shall be returned to the pit under the direction of the FOL. Backfill should be returned to the trench or test pit in 6 inch to 1 foot lifts and compacted with the bucket. Remote controlled tampers or rollers may be lowered into the trench and operated from top side. This procedure will continue to the grade surface. It is recommended that the trench be tracked or rolled in. During excavation, clean soil from the top 2 feet may have been separated to be used to cover the last segments. Where these materials are not clean, it is recommended that clean fill be used for the top cover.~~

- Soil type classification

7.0 REFERENCES

American Society for Testing and Materials, 1987. ASTM Standards D1587-83 and D1586-84. ASTM Annual Book of Standards. ASTM. Philadelphia, Pennsylvania. Volume 4.08.

NUS Corporation, 1986. Hazardous Material Handling Training Manual.

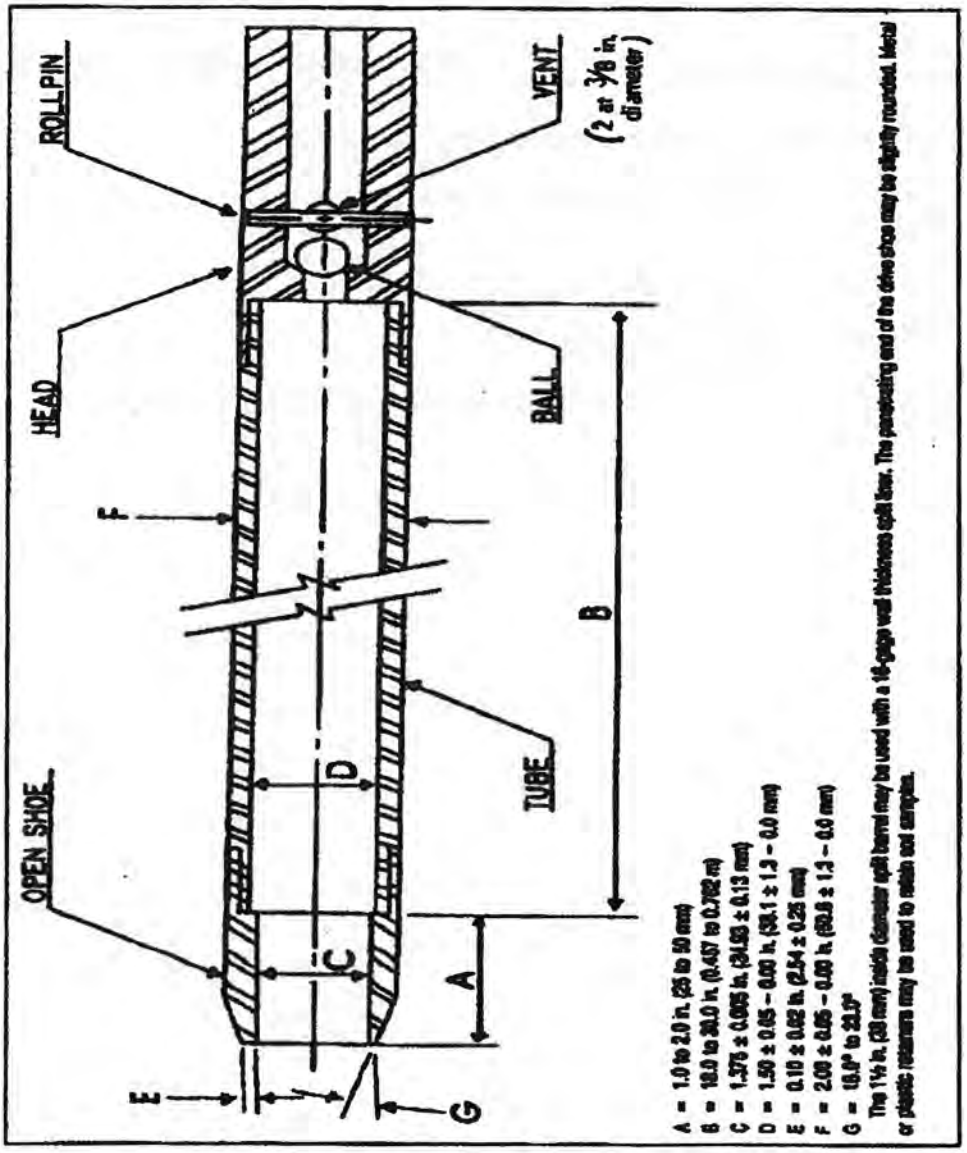
NUS Corporation and CH2M Hill, August, 1987. Compendium of Field Operation Methods. Prepared for the U.S. EPA.

OSHA, Excavation, Trenching and Shoring 29 CFR 1926.650-653.

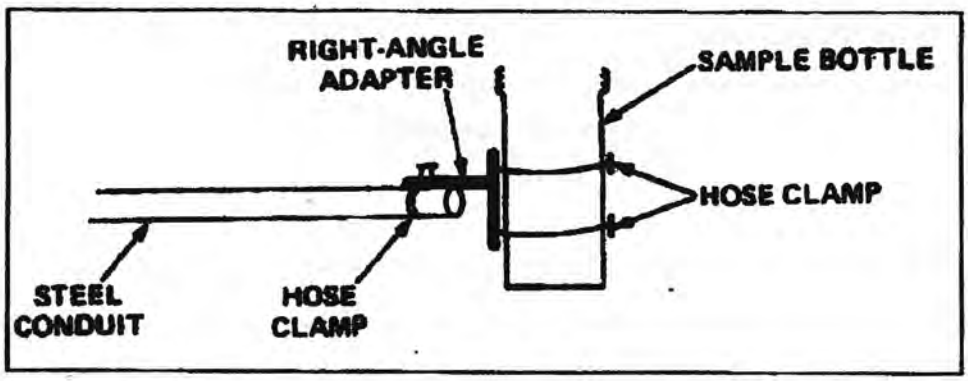
OSHA, Confined Space Entry 29 CFR 1910.146.

USEPA, November 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual.

ATTACHMENT B
SPLIT-SPOON SAMPLER



ATTACHMENT D
REMOTE SAMPLE HOLDER FOR TEST PIT/TRENCH SAMPLING



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		Effective Date 09/03	Revision 3
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject DIRECT PUSH TECHNOLOGY (GEOPROBE®/HYDROPUNCH™)		Approved D. Senovich	

TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this procedure is to provide general reference information on Direct Push Technology (DPT). DPT is designed to collect soil, groundwater, and soil gas samples without using conventional drilling techniques. The advantage of using DPT over conventional drilling includes the generation of little or no drill cuttings, sampling in locations with difficult accessibility, reduced overhead clearance requirements, no fluid introduction during probing, and typical lower costs per sample than with conventional techniques. Disadvantages include a maximum penetration depth of approximately 15 to 40 feet in dense soils (although it may be as much as 60 to 80 feet in certain types of geological environments), reduced capability of obtaining accurate water-level measurements, and the inability to install permanent groundwater monitoring wells. The methods and equipment described herein are for collection of surface and subsurface soil samples and groundwater samples. Soil gas sampling is discussed in SOP SA-2.4.

2.0 SCOPE

This procedure provides information on proper sampling equipment and techniques for DPT. Review of the information contained herein will facilitate planning of the field sampling effort by describing standard sampling techniques. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require adjustments in methodology.

3.0 GLOSSARY

Direct Push Technology (DPT) - DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools. A primary advantage of DPT over conventional drilling techniques is that DPT results in the generation of little or no investigation derived waste.

Geoprobe® - Geoprobe® is a manufacturer of a hydraulically-powered, percussion/probing machines utilizing DPT to collect subsurface environmental samples. Geoprobe® relies on a relatively small amount of static weight (vehicle) combined with percussion as the energy for advancement of a tool string. The Geoprobe® equipment can be mounted in a multitude of vehicles for access to all types of environmental sites.

HydroPunch™ - HydroPunch™ is a manufacturer of stainless steel and Teflon® sampling tools that are capable of collecting representative groundwater and/or soil samples without requiring the installation of a groundwater monitoring well or conventional soil boring. HydroPunch™ is an example of DPT sampling equipment.

Flame Ionization Detector (FID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing a flame as the energizing source.

Photo Ionization Detector (PID) - A portable instrument for the measurement of many combustible organic compounds and a few inorganic compounds in air at parts-per million levels. The basis for the detection is the ionization of gaseous species utilizing ultraviolet radiation as the energizing source.

4.0 RESPONSIBILITIES

Project Manager - The Project Manager is responsible for selecting and/or reviewing the appropriate DPT drilling procedure required to support the project objectives.

Field Operations Leader (FOL)- The FOL is primarily responsible for performing the DPT in accordance with the project-specific plan.

5.0 SOIL SAMPLING PROCEDURES

5.1 General

The common methodology for the investigation of the vadose zone is soil boring drilling and soil sampling. However, drilling soil borings can be very expensive. Generally the advantage of DPT for subsurface soil sampling is the reduced cost of disposal of drilling cuttings and shorter sampling times.

5.2 Sampling Equipment

Equipment needed for conducting DPT drilling for subsurface soil sampling includes, but is not limited to, the following:

- Geoprobe® Sampling Kit
- Cut-resistant gloves
- 4-foot x 1.5-inch diameter macrocore sampler
- Probe sampling adapters
- Roto-hammer with 1.5-inch bit
- Disposable acetate liners for soil macrocore sampler
- Cast aluminum or steel drive points
- Geoprobe® AT-660 Series Large Bore Soil Sampler, or equivalent
- Standard decontamination equipment and solutions

For health and safety equipment and procedures, follow the direction provided in the Safe Work Permit in Attachment 1, or the more detailed directions provided in the project's Health and Safety Plan.

5.3 DPT Sampling Methodology

There are several methods for the collection of soil samples using DPT drilling. The most common method is discussed in the following section. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project-specific plan.

- Macrocore samplers fitted with detachable aluminum or steel drive points are driven into the ground using hydraulic pressure. If there is concrete or pavement over a sampling location, a Roto-hammer is used to drill a minimum 1.5-inch diameter hole through the surface material. A Roto-hammer may also be used if very dense soils are encountered.

- The sampler is advanced continuously in 4-foot intervals or less if desired. No soil cuttings are generated because the soil which is not collected in the sampler is displaced within the formation.

- The sampler is retracted from the hole, and the 4-foot continuous sample is removed from the outer coring tube. The sample is contained within an inner acetate liner.

- Attach the metal trough from the Geoprobe® Sampling Kit firmly to the tail gate of a vehicle. If a vehicle with a tail gate is not available, secure the trough on another suitable surface.

- Place the acetate liner containing the soils in the trough.

- While wearing cut-resistant gloves (constructed of leather or other suitable material), cut the acetate liner through its entire length using the double-bladed knife that accompanies the Geoprobe® Sampling Kit. Then remove the strip of acetate from the trough to gain access to the collected soils. Do not attempt to cut the acetate liner while holding it in your hand.

- Field screen the sample with an FID or PID, and observe/examine the sample (according to SOP GH-1.3). If appropriate, transfer the sample to sample bottles for laboratory analysis. If additional volume is required, push an additional boring adjacent to the first and composite/mix the same interval. Field compositing is usually not acceptable for sample requiring volatile organics analysis.

- Once sampling has been completed, the hole is backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch is used to cap holes through paved or concrete areas. All holes should be finished smooth to existing grade.

- In the event the direct push van/truck cannot be driven to a remote location or a sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric operated equipment (e.g., jack hammer).

- Sampling equipment is decontaminated prior to collecting the next sample.

~~6.0 GROUNDWATER SAMPLING PROCEDURES~~

~~6.1 General~~

~~The most common methodology for the investigation of groundwater is the installation and sampling of permanent monitoring wells. If only groundwater screening is required, the installation and sampling of temporary well points may be performed. The advantage of temporary well point installation using DPT is reduced cost due to no or minimal disposal of drilling cuttings and well construction materials, and shorter installation/times sampling.~~

~~Two disadvantages of DPT drilling for well point installation are:~~

- ~~In aquifers with low yields, well points may have to be sampled without purging or development.~~
- ~~If volume requirements are high, this method can be time consuming for low yield aquifers.~~

~~6.2 Sampling Equipment~~

~~Equipment needed for temporary well installation and sampling using DPT includes, but is not limited, to the following:~~

- ~~2 foot x 1 inch diameter mill slotted (0.005 to 0.02 inch) well point~~
- ~~Connecting rods~~
- ~~Rote-hammer with 1.5 inch bit~~
- ~~Mechanical jack~~
- ~~1/4 inch OD polyethylene tubing~~
- ~~3/8 inch OD polyethylene tubing~~
- ~~Peristaltic pump~~
- ~~Standard decontamination equipment and solutions~~

6.3 ~~DPT Temporary Well Point Installation and Sampling Methodology~~

~~There are several methods for the installation and sampling of temporary well points using DPT. The most common methodology is discussed below. Variations of the following method may be conducted upon approval of the Project Manager in accordance with the project specific plan.~~

- ~~A 2-foot x 1-inch diameter mill-slotted (0.005 to 0.02-inch) well point attached to connecting rods is driven into the ground to the desired depth using a rotary electric hammer or other direct push drill rig. If there is concrete or pavement over a sampling location, a Roto-hammer or electric coring machine is used to drill a hole through the surface material.~~
- ~~The well point will be allowed to equilibrate for at least 15 minutes, after which a measurement of the static water level will be taken. The initial measurement of the water level will be used to assess the amount of water which is present in the well point and to determine the amount of silt and sand infiltration that may have occurred.~~
- ~~The well point will be developed using a peristaltic pump and polyethylene tubing to remove silt and sand which may have entered the well point. The well point is developed by inserting polyethylene tubing to the bottom of the well point and lifting and lowering the tubing slightly while the pump is operating. The pump will be operated at a maximum rate of approximately 2 liters per minute. After removal of sediment from the bottom of the well point, the well point will be vigorously pumped at maximum capacity until discharge water is visibly clear and no further sediments are being generated. Measurements of pH, specific conductance, temperature, and turbidity shall be recorded every 5 to 10 minutes during the purging process. After two consistent readings of pH, specific conductance, temperature and turbidity (±10 percent), the well may be sampled.~~
- ~~A sample will be collected using the peristaltic pump set at the same or reduced speed as during well development. Samples (with the exception of the samples to be analyzed for volatile organic compounds, VOCs) will be collected directly from the pump discharge. Sample containers for VOCs will be filled by (first shutting off the pump) crimping the discharge end of the sample tubing when filled, removing the inlet end of the sample tubing from the well, suspending the inlet tubing above the vial, and allowing water to fill each vial by gravity flow.~~
- ~~Once the groundwater sample has been collected, the connecting rods and well point will be removed from the hole with the direct push rig hydraulics. The hole will be backfilled with bentonite chips or bentonite cement grout, depending upon project requirements. Asphalt or concrete patch will be used to cap holes through paved or concrete areas. All holes will be finished smooth to existing grade.~~
- ~~In the event the direct push van/truck cannot be driven to a remote location or sampling location with difficult accessibility, sampling probes may be advanced and sampled manually or with air/electric operated equipment (e.g., jack hammer).~~
- ~~Decontaminate the equipment before moving to the next location.~~

7.0 RECORDS

A record of all field procedures, tests, and observations must be recorded in the field logbook, boring logs, and sample log sheets, as needed. Entries should include all pertinent data regarding the investigation. The use of sketches and field landmarks will help to supplement the investigation and evaluation.

ATTACHMENT 1
SAFE WORK PERMIT FOR DPT OPERATIONS

Permit No. _____ Date: _____ Time: From _____ to _____

SECTION I: General Job Scope

I. Work limited to the following (description, area, equipment used): **Monitoring well drilling and installation through direct push technology**

II. Required Monitoring Instruments: _____

III. Field Crew: _____

IV. On-site Inspection conducted ☐ Yes ☐ No Initials of Inspector _____ _____
TtNUS

SECTION II: General Safety Requirements (To be filled in by permit issuer)

V. Protective equipment required
Level D ☒ Level B ☐
Level C ☐ Level A ☐
Detailed on Reverse

Respiratory equipment required

Full face APR	☐	Escape Pack	☐
Half face APR	☐	SCBA	☐
SKA-PAC SAR	☐	Bottle Trailer	☐
Skid Rig	☐	None	☒

Level D Minimum Requirements: Sleeved shirt and long pants, safety footwear, and work gloves. Safety glasses, hard hats, and hearing protection will be worn when working near or sampling in the vicinity of the DPT rig.

Modifications/Exceptions.

VI. Chemicals of Concern _____ Action Level(s) _____ Response Measures _____

VII. Additional Safety Equipment/Procedures

Hard-hat	☒ Yes ☐ No	Hearing Protection (Plugs/Muffs)	☒ Yes ☐ No
Safety Glasses	☒ Yes ☐ No	Safety belt/harness	☐ Yes ☒ No
Chemical/splash goggles	☐ Yes ☒ No	Radio	☐ Yes ☒ No
Splash Shield	☐ Yes ☒ No	Barricades	☒ Yes ☐ No
Splash suits/coveralls	☐ Yes ☐ No	Gloves (Type - _____)	☐ Yes ☐ No
Steel toe Work shoes or boots	☒ Yes ☐ No	Work/warming regimen	☐ Yes ☐ No

Modifications/Exceptions: Reflective vests for high traffic areas.

VIII. Procedure review with permit acceptors

	Yes	NA		Yes	NA
Safety shower/eyewash (Location & Use)	☐	☒	Emergency alarms	☐	☐
Daily tail gate meetings	☒	☐	Evacuation routes	☐	☐
Contractor tools/equipment/PPE inspected	☐	☐	Assembly points	☐	☐

IX. Site Preparation

Utility Clearances obtained for areas of subsurface investigation	☐ Yes	☐ No
Physical hazards removed or blockaded	☐ Yes	☐ No
Site control boundaries demarcated/signage	☐ Yes	☐ No

X. Equipment Preparation

	Yes	NA
Equipment drained/depressurized	☐	☒
Equipment purged/cleaned	☐	☒
Isolation checklist completed	☐	☒
Electrical lockout required/field switch tested	☐	☒
Blinds/misalignments/blocks & bleeds in place	☐	☒
Hazardous materials on walls/behind liners considered	☐	☒

XI. Additional Permits required (Hot work, confined space entry). ☐ Yes ☐ No
If yes, complete permit required or contact Health Sciences, Pittsburgh Office

XII. Special instructions, precautions:

Permit Issued by: _____ Permit Accepted by: _____

TETRA TECH NUS, INC.	STANDARD OPERATING PROCEDURES	Number SA-6.1	Page 1 of 11
		Effective Date 02/04	Revision 3
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject NON-RADIOLOGICAL SAMPLE HANDLING		Approved D. Senovich	

TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide information on sample preservation, packaging, and shipping procedures to be used in handling environmental samples submitted for chemical constituent, biological, or geotechnical analysis. Sample chain-of-custody procedures and other aspects of field documentation are addressed in SOP SA-6.3. Sample identification is addressed in SOP CT-04.

2.0 SCOPE

This procedure describes the appropriate containers to be used for samples depending on the analyses to be performed, and the steps necessary to preserve the samples when shipped off site for chemical analysis.

3.0 GLOSSARY

Hazardous Material - A substance or material which has been determined by the Secretary of Transportation to be capable of posing an unreasonable risk to health, safety, and property when transported in commerce, and which has been so designated. Under 49 CFR, the term includes hazardous substances, hazardous wastes, marine pollutants, and elevated temperature materials, as well as materials designated as hazardous under the provisions of §172.101 and §172.102 and materials that meet the defining criteria for hazard classes and divisions in Part 173. With slight modifications, IATA has adopted DOT "hazardous materials" as IATA "Dangerous Goods."

Hazardous Waste - Any substance listed in 40 CFR, Subpart D (y261.30 et seq.), or otherwise characterized as ignitable, corrosive, reactive, or toxic (as defined by Toxicity Characteristic Leaching Procedure, TCLP, analysis) as specified under 40 CFR, Subpart C (y261.20 et seq.), that would be subject to manifest requirements specified in 40 CFR 262. Such substances are defined and regulated by EPA.

Marking - A descriptive name, identification number, instructions, cautions, weight, specification or UN marks, or combination thereof required on outer packaging of hazardous materials.

n.o.i - Not otherwise indicated (may be used interchangeably with n.o.s.).

n.o.s. - Not otherwise specified.

Packaging - A receptacle and any other components or materials necessary for compliance with the minimum packaging requirements of 49 CFR 174, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank-car tanks to perform a containment function in conformance with the minimum packaging requirements of 49 CFR 173.24(a) & (b).

Placard - Color-coded, pictorial sign which depicts the hazard class symbol and name and which is placed on the side of a vehicle transporting certain hazardous materials.

Common Preservatives:

- Hydrochloric Acid - HCl
- Sulfuric Acid - H_2SO_4
- Nitric Acid - HNO_3
- Sodium Hydroxide - NaOH

Other Preservatives

- Zinc Acetate
- Sodium Thiosulfate - $Na_2S_2O_3$

Normality (N) - Concentration of a solution expressed as equivalent per liter, an equivalent being the amount of a substance containing 1 gram-atom of replaceable hydrogen or its equivalent.

Reportable Quantity (RQ) - For the purposes of this SOP, means the quantity specified in column 3 of the Appendix to DOT 49 CFR §172.101 for any material identified in column 1 of the appendix. A spill greater than the amount specified must be reported to the National Response Center.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the location and time of collection.

4.0 RESPONSIBILITIES

Field Operations Leader - Directly responsible for the bottling, preservation, labeling, packaging, shipping, and custody of samples up to and including release to the shipper.

Field Samplers - Responsible for initiating the Chain-of-Custody Record (per SOP SA-6.3), implementing the packaging and shipping requirements, and maintaining custody of samples until they are relinquished to another custodian or to the shipper.

5.0 PROCEDURES

Sample identification, labeling, documentation, and chain-of-custody are addressed by SOP SA-6.3.

5.1 Sample Containers

Different types of chemicals react differently with sample containers made of various materials. For example, trace metals adsorb more strongly to glass than to plastic, whereas many organic chemicals may dissolve various types of plastic containers. Attachments A and B show proper containers (as well as other information) per 40 CFR 136. In general, the sample container shall allow approximately 5-10 percent air space ("ullage") to allow for expansion/vaporization if the sample warms during transport. However, for collection of volatile organic compounds, head space shall be omitted. The analytical laboratory will generally provide certified-clean containers for samples to be analyzed for chemical constituents. Shelby tubes or other sample containers are generally provided by the driller for samples requiring geotechnical analysis. Sufficient lead time shall be allowed for a delivery of sample container orders. Therefore, it is critical to use the correct container to maintain the integrity of the sample prior to analysis.

Once opened, the container must be used at once for storage of a particular sample. Unused but opened containers are to be considered contaminated and must be discarded. Because of the potential for introduction of contamination, they cannot be reclosed and saved for later use. Likewise, any unused containers which appear contaminated upon receipt, or which are found to have loose caps or a missing Teflon liner (if required for the container), shall be discarded.

5.2 Sample Preservation

Many water and soil samples are unstable and therefore require preservation to prevent changes in either the concentration or the physical condition of the constituent(s) requiring analysis. Although complete and irreversible preservation of samples is not possible, preservation does retard the chemical and biological

changes that inevitably take place after the sample is collected. Preservation techniques are usually limited to pH control, chemical addition(s), and refrigeration/ freezing (certain biological samples only).

5.2.1 Overview

The preservation techniques to be used for various analytes are listed in Attachments A and B. Reagents required for sample preservation will either be added to the sample containers by the laboratory prior to their shipment to the field or be added in the field (in a clean environment). Only high purity reagents shall be used for preservation. In general, aqueous samples of low-concentration organics (or soil samples of low- or medium-concentration organics) are cooled to 4°C. Medium-concentration aqueous samples, high-hazard organic samples, and some gas samples are typically not preserved. Low-concentration aqueous samples for metals are acidified with HNO_3, whereas medium-concentration and high-hazard aqueous metal samples are not preserved. Low- or medium-concentration soil samples for metals are cooled to 4°C, whereas high-hazard samples are not cooled.

The following subsections describe the procedures for preparing and adding chemical preservatives. Attachments A and B indicate the specific analytes which require these preservatives.

The FOL is responsible for ensuring that an accurate Chemical Inventory is created and maintained for all hazardous chemicals brought to the work site (see Section 5 of the TtNUS Health and Safety Guidance Manual). Furthermore, the FOL must ensure that a corresponding Material Safety Data Sheet (MSDS) is collected for every substance entered on the site Chemical Inventory, and that all persons using/handling/ disposing of these substances review the appropriate MSDS for substances they will work with. The Chemical Inventory and the MSDSs must be maintained at each work site in a location and manner where they are readily-accessible to all personnel.

5.2.2 Preparation and Addition of Reagents

Addition of the following acids or bases may be specified for sample preservation; these reagents shall be analytical reagent (AR) grade or purer and shall be diluted to the required concentration with deionized water before field sampling commences. To avoid uncontrolled reactions, be sure to Add Acid to water (not vice versa). A dilutions guide is provided below.

Acid/Base	Dilution	Concentration	Estimated Amount Required for Preservation
Hydrochloric Acid (HCl)	1 part concentrated HCl: 1 part double-distilled, deionized water	6N	5-10 mL
Sulfuric Acid (H_2SO_4)	1 part concentrated H_2SO_4: 1 part double-distilled, deionized water	18N	2 - 5 mL
Nitric Acid (HNO_3)	Undiluted concentrated HNO_3	16N	2 - 5 mL
Sodium Hydroxide (NaOH)	400 grams solid NaOH dissolved in 870 mL double-distilled, deionized water; yields 1 liter of solution	10N	2 mL

The amounts required for preservation shown in the above table assumes proper preparation of the preservative and addition of the preservative to one liter of aqueous sample. This assumes that the sample is initially at pH 7, is poorly buffered, and does not contain particulate matter; as these conditions vary, more preservative may be required. Consequently, the final sample pH must be checked using narrow-range pH paper, as described in the generalized procedure detailed below:

- Pour off 5-10 mL of sample into a dedicated, clean container. Use some of this sample to check the initial sample pH using wide range (0-14) pH paper. Never dip the pH paper into the sample; always apply a drop of sample to the pH paper using a clean stirring rod or pipette.

- Add about one-half of the estimated preservative required to the original sample bottle. Cap and invert gently several times to mix. Check pH (as described above) using medium range pH paper (pH 0-6 or pH 7.5-14, as applicable).

- Cap sample bottle and seal securely.

Additional considerations are discussed below:

- To test if ascorbic acid must be used to remove oxidizing agents present in the sample before it can be properly preserved, place a drop of sample on KI-starch paper. A blue color indicates the need for ascorbic acid addition.

  If required, add a few crystals of ascorbic acid to the sample and retest with the KI-starch paper. Repeat until a drop of sample produces no color on the KI-starch paper. Then add an additional 0.6 grams of ascorbic acid per each liter of sample volume.

  Continue with proper base preservation of the sample as described above.

- Samples for sulfide analysis must be treated by the addition of 4 drops (0.2 mL) of 2N zinc acetate solution per 100 ml of sample.

  The 2N zinc acetate solution is made by dissolving 220 grams of zinc acetate in 870 mL of double-distilled, deionized water to make 1 liter of solution.

  The sample pH is then raised to 9 using the NaOH preservative.

- Sodium thiosulfate must be added to remove residual chlorine from a sample. To test the sample for residual chlorine use a field test kit specially made for this purpose.

  If residual chlorine is present, add 0.08 grams of sodium thiosulfate per liter of sample to remove the residual chlorine.

  Continue with proper acidification of the sample as described above.

For biological samples, 10% buffered formalin or isopropanol may also be required for preservation. Questions regarding preservation requirements should be resolved through communication with the laboratory before sampling begins.

5.3 Field Filtration

At times, field-filtration may be required to provide for the analysis of dissolved chemical constituents. Field-filtration must be performed prior to the preservation of samples as described above. General procedures for field filtration are described below:

- The sample shall be filtered through a non-metallic, 0.45-micron membrane filter, immediately after collection. The filtration system shall consist of dedicated filter canister, dedicated tubing, and a peristaltic pump with pressure or vacuum pumping squeeze action (since the sample is filtered by mechanical peristalsis, the sample travels only through the tubing).

- To perform filtration, thread the tubing through the peristaltic pump head. Attach the filter canister to the discharge end of the silicon tubing (note flow direction arrow); attach the aqueous sample container to the intake end of the silicon tubing. Turn the peristaltic pump on and perform filtration. Run approximately 100 ml of sample through the filter and discard prior to sample collection.

- Continue by preserving the filtrate (contained in the filter canister), as applicable and generally described above.

5.4 Sample Packaging and Shipping

Only employees who have successfully completed the TtNUS "Shipping Hazardous Materials" training course are authorized to package and ship hazardous substances. These trained individuals are responsible for performing shipping duties in accordance with this training.

Samples collected for shipment from a site shall be classified as either environmental or hazardous material samples. Samples from drums containing materials other than Investigative Derived Waste (IDW) and samples obtained from waste piles or bulk storage tanks are generally shipped as hazardous materials. A distinction must be made between the two types of samples in order to:

- Determine appropriate procedures for transportation of samples (if there is any doubt, a sample shall be considered hazardous and shipped accordingly.)

- Protect the health and safety of transport and laboratory personnel receiving the samples (special precautions are used by the shipper and at laboratories when hazardous materials are received.)

Detailed procedures for packaging environmental samples are outlined in the remainder of this section.

5.4.1 Environmental Samples

Environmental samples are packaged as follows:

- Place properly identified sample container, with lid securely fastened, in a plastic bag (e.g. Ziploc baggie), and seal the bag.

- Place sample in a cooler constructed of sturdy material which has been lined with a large, plastic bag (e.g. "garbage" bag). Drain plugs on coolers must be taped shut.

- Pack with enough cushioning materials such as bubble wrap (shoulders of bottles must be iced if required) to minimize the possibility of the container breaking.

- If cooling is required (see Attachments A and B), place ice around sample container shoulders, and on top of packing material (minimum of 8 pounds of ice for a medium-size cooler).

- Seal (i.e., tape or tie top in knot) large liner bag.

- The original (top, signed copy) of the COC form shall be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one COC form, the COC form should be sent with the cooler containing the vials for VOC analysis. The COC form should then state how many coolers are included with that shipment.

- Close and seal outside of cooler as described in SOP SA-6.3. Signed custody seals must be used.

Coolers must be marked as containing "Environmental Samples." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT marking or labeling is required; there are no DOT restrictions on mode of transportation.

6.0 REFERENCES

American Public Health Association, 1981. Standard Methods for the Examination of Water and Wastewater, 15th Edition. APHA, Washington, D.C.

International Air Transport Association (latest issue). Dangerous Goods Regulations, Montreal, Quebec, Canada.

U.S. Department of Transportation (latest issue). Hazardous Materials Regulations, 49 CFR 171-177.

U.S. EPA, 1984. "Guidelines Establishing Test Procedures for the Analysis of Pollutants under Clean Water Act." Federal Register, Volume 49 (209), October 26, 1984, p. 43234.

U.S. EPA, 1979. Methods for Chemical Analysis of Water and Wastes. EPA-600/4-79-020, U.S. EPA-EMSL, Cincinnati, Ohio.

ATTACHMENT A

GENERAL SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

Sample Type and Concentration		Container[1]	Sample Size	Preservation[2]	Holding Time[2]
WATER					
Organics (GC&GC/MS)	VOC Low	Borosilicate glass	2 x 40 mL	Cool to 4°C HCl to ≤ 2	14 days[9]
	Extractables (Low SVOCs and pesticide/PCBs)	Amber glass	2x2 L or 4x1 L	Cool to 4°C	7 days to extraction; 40 days after extraction
	Extractables (Medium SVOCs and pesticide/PCBs)	Amber glass	2x2 L or 4x1 L	None	7 days to extraction; 40 days after extraction
Inorganics	Metals Low	High-density polyethylene	1 L	HNO_3 to pH ≤2	6 months (Hg-28 days)
	Medium	Wide-mouth glass	16 oz.	None	6 months
	Cyanide Low	High-density polyethylene	1 L	NaOH to pH>12	14 days
	Cyanide Medium	Wide-mouth glass	16 oz.	None	14 days
Organic/ Inorganic	High Hazard	Wide-mouth glass	8 oz.	None	14 days
SOIL					
Organics (GC&GC/MS)	VOC	EnCore Sampler	(3) 5 g Samplers	Cool to 4°C	48 hours to lab preservation
	Extractables (Low SVOCs and pesticides/PCBs)	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
	Extractables (Medium SVOCs and pesticides/PCBs)	Wide-mouth glass	8 oz.	Cool to 4°C	14 days to extraction; 40 days after extraction
Inorganics	Low/Medium	Wide-mouth glass	8 oz.	Cool to 4°C	6 months (Hg - 28 days) Cyanide (14 days)
Organic/Inorganic	High Hazard	Wide-mouth glass	8 oz.	None	NA
Dioxin/Furan	All	Wide-mouth glass	4 oz.	None	35 days until extraction; 40 days after extraction
TCLP	All	Wide-mouth glass	8 oz.	None	7 days until preparation; analysis as per fraction
AIR					
Volatile Organics	Low/Medium	Charcoal tube -- 7 cm long, 6 mm OD, 4 mm ID	100 L air	Cool to 4°C	5 days recommended

1 All glass containers should have Teflon cap liners or septa.
2 See Attachment E. Preservation and maximum holding time allowances per 40 CFR 136.

ATTACHMENT B

ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS:			
Acidity	P, G	Cool, 4°C	14 days
Alkalinity	P, G	Cool, 4°C	14 days
Ammonia - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Biochemical Oxygen Demand (BOD)	P, G	Cool, 4°C	48 hours
Bromide	P, G	None required	28 days
Chemical Oxygen Demand (COD)	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Chloride	P, G	None required	28 days
Chlorine, Total Residual	P, G	None required	Analyze immediately
Color	P, G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination	P, G	Cool, 4°C; NaOH to pH 12; 0.6 g ascorbic acid[5]	14 days[6]
Fluoride	P	None required	28 days
Hardness	P, G	HNO_3 to pH 2; H_2SO_4 to pH 2	6 months
Total Kjeldahl and Organic Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrate - Nitrogen	P, G	None required	48 hours
Nitrate-Nitrite - Nitrogen	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Nitrite - Nitrogen	P, G	Cool, 4°C	48 hours
Oil & Grease	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Total Organic Carbon (TOC)	P, G	Cool, 4°C; HCl or H_2SO_4 to pH 2	28 days
Orthophosphate	P, G	Filter immediately; Cool, 4°C	48 hours
Oxygen, Dissolved-Probe	G Bottle & top	None required	Analyze immediately
Oxygen, Dissolved-Winkler	G Bottle & top	Fix on site and store in dark	8 hours
Phenols	G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Phosphorus, Total	P, G	Cool, 4°C; H_2SO_4 to pH 2	28 days
Residue, Total	P, G	Cool, 4°C	7 days
Residue, Filterable (TDS)	P, G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS)	P, G	Cool, 4°C	7 days
Residue, Settleable	P, G	Cool, 4°C	48 hours
Residue, Volatile (Ash Content)	P, G	Cool, 4°C	7 days
Silica	P	Cool, 4°C	28 days
Specific Conductance	P, G	Cool, 4°C	28 days
Sulfate	P, G	Cool, 4°C	28 days

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE TWO

Parameter Number/Name	Container[1]	Preservation[2][3]	Maximum Holding Time[4]
INORGANIC TESTS (Cont'd):			
Sulfide	P, G	Cool, 4°C; add zinc acetate plus sodium hydroxide to pH 9	7 days
Sulfite	P, G	None required	Analyze immediately
Turbidity	P, G	Cool, 4°C	48 hours
METALS:[7]			
Chromium VI (Hexachrome)	P, G	Cool, 4°C	24 hours
Mercury (Hg)	P, G	HNO_3 to pH 2	28 days
Metals, except Chromium VI and Mercury	P, G	HNO_3 to pH 2	6 months
ORGANIC TESTS:[8]			
Purgeable Halocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	14 days
Purgeable Aromatic Hydrocarbons	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] HCl to pH 2[9]	14 days
Acrolein and Acrylonitrile	G, Teflon-lined septum	Cool, 4°C; 0.008% $Na_2S_2O_3$[5] adjust pH to 4-5[10]	14 days
Phenols[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Benzidines[11],[12]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction[13]
Phthalate esters[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitrosamines[11],[14]	G, Teflon-lined cap	Cool, 4°C; store in dark; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
PCBs[11]	G, Teflon-lined cap	Cool, 4°C	7 days until extraction; 40 days after extraction
Nitroaromatics & Isophorone[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Polynuclear Aromatic Hydrocarbons (PAHs)[11],[14]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]; store in dark	7 days until extraction; 40 days after extraction
Haloethers[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction
Dioxin/Furan (TCDD/TCDF)[11]	G, Teflon-lined cap	Cool, 4°C; 0.008% $Na_2S_2O_3$[5]	7 days until extraction; 40 days after extraction

ATTACHMENT B
ADDITIONAL REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES
PAGE THREE

(1) Polyethylene (P): generally 500 ml or Glass (G): generally 1L.
(2) Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.
(3) When any sample is to be shipped by common carrier or sent through the United States Mail, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR Part 172).
(4) Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer periods, and has received a variance from the Regional Administrator.
(5) Should only be used in the presence of residual chlorine.
(6) Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustments are made to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.
(7) Samples should be filtered immediately on site before adding preservative for dissolved metals.
(8) Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.
(9) Sample receiving no pH adjustment must be analyzed within 7 days of sampling.
(10) The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.
(11) When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9; samples preserved in this manner may be held for 7 days before extraction and for 40 days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re: the requirement for thiosulfate reduction of residual chlorine) and footnotes 12, 13 (re: the analysis of benzidine).
(12) If 1,2-diphenylthydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.
(13) Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.
(14) For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7-10 with NaOH within 24 hours of sampling.
(15) The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

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		Effective Date 03/09/09	Revision 3
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject FIELD DOCUMENTATION		Approved Tom Johnston	

TABLE OF CONTENTS

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to identify and designate the field data record forms, logs, and reports generally initiated and maintained for documenting Tetra Tech NUS, Inc. (TtNUS) field activities.

2.0 SCOPE

Documents presented within this SOP (or equivalents) shall be used for all TtNUS field activities, as applicable. Other or additional documents may be required by specific client contracts or project planning documents.

3.0 GLOSSARY

None.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager (PM) - The PM is responsible for obtaining hardbound controlled-distribution logbooks (from the appropriate source), as needed. In addition, the Project Manager is responsible for placing all field documentation used in site activities (i.e., records, field reports, sample data sheets, field notebooks, and the site logbook) in the project's central file upon the completion of field work.

Field Operations Leader (FOL) - The FOL is responsible for ensuring that the site logbook, notebooks, and all appropriate and current forms and field reports included in this SOP (and any additional forms required by the contract) are correctly used, accurately filled out, and completed in the required time frame.

General personnel qualifications for field documentation activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate procedures for documentation, handling, packaging, and shipping.

5.0 PROCEDURES

5.1 SITE LOGBOOK

5.1.1 General

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major on-site activities are documented. At a minimum, record or reference the following activities/events (daily) in the site logbook:

- All field personnel present
- Arrival/departure times and names of site visitors
- Times and dates of health and safety training
- Arrival/departure times of equipment
- Times and dates of equipment calibration

- Start and/or completion of borehole, trench, monitoring well installation activities, etc.
- Daily on-site activities
- Sample pickup information
- Health and safety issues (level of protection, personal protective equipment [PPE], etc.)
- Weather conditions

Maintain a site logbook for each project and initiate it at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Make entries every day that on-site activities take place involving TtNUS or subcontractor personnel. Upon completion of the fieldwork, provide the site logbook to the PM or designee for inclusion in the project's central file.

Record the following information on the cover of each site logbook:

- Project name
- TtNUS project number
- Sequential book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks (see Section 5.2) but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). An example of a typical site logbook entry is shown in Attachment A.

If measurements are made at any location, either record the measurements and equipment used in the site logbook or reference the field notebook in which the measurements are recorded (see Attachment A).

Make all logbook, notebook, and log sheet entries in indelible ink (black pen is preferred). No erasures are permitted. If an incorrect entry is made, cross out the entry with a single strike mark, initial, and date it. At the completion of entries by any individual, the logbook pages used must be signed and dated by the person making the entries. The site logbook must also be signed by the FOL at the end of each day.

5.1.2 Photographs

Sequentially number movies, slides, or photographs taken of a site or any monitoring location to correspond to logbook/notebook entries. Enter the name of the photographer, date, time, site location, site description, and weather conditions in the logbook/notebook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. However, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook/notebook. If possible, such techniques shall be avoided because they can adversely affect the accuracy of photographs. Chain-of-custody procedures depend on the subject matter, type of camera (digital or film), and the processing it requires. Follow chain-of-custody procedures for film used for aerial photography, confidential information, or criminal investigation. After processed, consecutively number the slides of photographic prints and label them according to the logbook/notebook descriptions. Docket the site photographs and associated negatives and/or digitally saved images to compact disks into the project's central file.

5.2 FIELD NOTEBOOKS

Key field team personnel may maintain a separate dedicated field notebook to document the pertinent field activities conducted directly under their supervision. For example, on large projects with multiple investigative sites and varying operating conditions, the Health and Safety Officer may elect to maintain a

separate field notebook. Where several drill rigs are in operation simultaneously, each site geologist assigned to oversee a rig must maintain a field notebook.

5.3 FIELD FORMS

All TtNUS field forms (see list in Section 6.0 of this SOP) can be found on the company's intranet site (http://intranet.ttnus.com) under Field Log Sheets. Forms may be altered or revised for project-specific needs, subject to client approval. Care must be taken to ensure that all essential information can be documented. Guidelines for completing these forms can be found in the related sampling SOPs.

5.3.1 Sample Collection, Labeling, Shipment, Request for Analysis, and Field Test Results

5.3.1.1 Sample Log Sheet

Sample log sheets are used to record specified types of data while sampling. The data recorded on these sheets are useful in describing the sample as well as pointing out any problems, difficulties, or irregularities encountered during sampling. Complete a sample log sheet for each sample obtained, including field quality control (QC) samples.

5.3.1.2 Sample Label

A typical sample label is illustrated in Attachment B. Complete the required information on the adhesive labels and apply them to every sample container. Obtain sample labels from the appropriate program/project source, request that they be electronically generated in house, or request them the laboratory subcontractor.

5.3.1.3 Chain-of-Custody Record

The chain-of-custody record is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as they are transferred from person to person. This form must be used as follows for any samples collected for chemical or geotechnical analysis whether the analyses are performed on site or off site:

- Retain one carbonless copy of the completed chain-of custody form in the field.
- Send one copy is sent to the PM (or designee)
- Send the original to the laboratory with the associated samples. Place the original (top, signed copy) of the chain-of custody form inside a large Ziploc®-type bag taped inside the lid of the shipping cooler. If multiple coolers are sent but are included on one chain-of custody form, send the form with the cooler containing vials for volatile organic compound (VOC) analysis or the cooler with the air bill attached. Indicate on the air bill how many coolers are included with that shipment.

An example of a chain-of-custody form is provided as Attachment C. After the samples are received at the laboratory, the sample cooler and contents are checked and any problems are noted on the enclosed chain-of custody form (any discrepancies between the sample labels and chain-of custody form and any other problems that are noted are resolved through communication between the laboratory point-of-contact and the TtNUS PM). The chain-of custody form is signed and copied. The laboratory will retain the copy, and the original becomes part of the samples' corresponding analytical data package.

5.3.1.4 Chain-of-Custody Seal

Attachment D is an example of a custody seal. The custody seal is an adhesive-backed label that is part of a chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transport to the laboratory. Sign and date custody seals

and affix them across the lid and body of each cooler (front and back) containing environmental samples (see SOP SA-6.1). Obtain custody seals from the laboratory (if available) or purchase them from a supplier.

5.3.1.5 Geochemical Parameters Log Sheets

Complete Field Analytical Log Sheets to record geochemical and/or natural attenuation field test results.

5.3.2 Hydrogeological and Geotechnical Forms

5.3.2.1 Groundwater Level Measurement Sheet

Complete a Groundwater Level Measurement Sheet for each round of water level measurements made at a site.

5.3.2.2 ~~Data Sheet for Pumping Test~~

~~During the performance of a pumping test (or an in-situ hydraulic conductivity test), a large amount of data must be recorded, often within a short time period. Use a Pumping Test Data Sheet to facilitate this task by standardizing the data collection format for the pumping well and observation wells, and allowing the time interval for collection to be established in advance.~~

5.3.2.3 ~~Packer Test Report Form~~

~~Complete a Packer Test Report Form for each well at which a packer test is conducted.~~

5.3.2.4 Boring Log

Complete a Summary Log of Boring, or Boring Log for each soil boring performed to document the materials encountered, operation and driving of casing, and locations/depths of samples collected. In addition, if volatile organics are monitored on cores, samples, cuttings from the borehole, or breathing zone, (using a photoionization detector [PID] or flame ionization detector [FID]), enter these readings on the boring log at the appropriate depth. When they become available, enter the laboratory sample number, concentrations of key contaminants, or other pertinent information in the "Remarks" column. This feature allows direct comparison of contaminant concentrations with soil characteristics.

5.3.2.5 ~~Monitoring Well Construction Details Form~~

~~Complete a Monitoring Well Construction Details Form for every monitoring well, piezometer, or temporary well point installed. This form contains specific information on length and type of well riser pipe and screen, backfill, filter pack, annular seal and grout characteristics, and surface seal characteristics. This information is important in evaluating the performance of the monitoring well, particularly in areas where water levels show temporal variation or where there are multiple (immiscible) phases of contaminants. Depending on the type of monitoring well (in overburden or bedrock, stick up or flush mount), different forms are used.~~

5.3.2.6 ~~Test Pit Log~~

~~When a test pit or trench is constructed for investigative or sampling purposes, a Test Pit Log must be filled out by the responsible field geologist or sampling technician.~~

5.3.2.7 Miscellaneous Monitoring Well Forms

Miscellaneous monitoring well forms that may be required on a project-specific basis include the Monitoring Well Materials Certificate of Conformance and Monitoring Well Development Record. Use a Monitoring Well Materials Certificate of Conformance to document all materials utilized during each monitoring well installation. Use a Monitoring Well Development Record to document all well development activities.

5.3.2.8 Miscellaneous Field Forms – Quality Assurance and Checklists

Miscellaneous field forms/checklists forms that may be required on a project-specific basis include the following:

- Container Sample and Inspection Sheet – use this form when a container (drum, tank, etc.) is sampled and/or inspected.
- QA Sample Log Sheet – use this form when a QA sample such as an equipment rinsate blank, source blank, etc. is collected.
- Field Task Modification Request (FTMR) – use this form to document deviations from the project planning documents. The FOL is responsible for initiating the FTMRs. Maintain copies of all FTMRs with the on-site planning documents, and place originals in the final evidence file.
- Field Project Daily Activities Checklist and Field Project Pre-Mobilization Checklist – used these during both the planning and field effort to ensure that all necessary tasks are planned for and completed. These two forms are not requirements but are useful tools for most field work.

5.3.3 Equipment Calibration and Maintenance Form

The calibration or standardization of monitoring, measuring, or test equipment is necessary to ensure the proper operation and response of the equipment, to document the accuracy, precision, or sensitivity of the measurements, and determine if correction should be applied to the readings. Some items of equipment require frequent calibration, others infrequent. Some are calibrated by the manufacturer, others by the user.

Each instrument requiring calibration has its own Equipment Calibration Log, which documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. Maintain an Equipment Calibration Log for each electronic measuring device used in the field; make entries for each day the equipment is used or in accordance with manufacturer recommendations.

5.4 FIELD REPORTS

The primary means of recording on-site activities is the site logbook. Other field notebooks may also be maintained. These logbooks and notebooks (and supporting forms) contain detailed information required for data interpretation or documentation but are not easily used for tracking and reporting of progress. Furthermore, the field logbook/notebooks remain on site for extended periods of time and are thus not accessible for timely review by project management. Other reports useful for tracking and reporting the progress of field activities are described below.

5.4.1 Daily Activities Report

To provide timely oversight of on-site contractors, complete and submit Daily Activities Reports (DARs) as described below.

5.4.1.1 Description

The DAR documents the activities and progress for each day's field work. Complete this report on a daily basis whenever there are drilling, test pitting, well construction, or other related activities occurring that involve subcontractor personnel. These sheets summarize the work performed and form the basis of payment to subcontractors. The DAR form can be found on the TtNUS Intranet site.

5.4.1.2 Responsibilities

It is the responsibility of the rig geologist to complete the DAR and obtain the driller's signature acknowledging that the times and quantities of material entered are correct.

5.4.1.3 Submittal and Approval

At the end of the shift, the rig geologist must submit the DAR to the FOL for review and filing. The Daily Activities Report is not a formal report and thus requires no further approval. The DARs are retained by the FOL for use in preparing the site logbook and in preparing weekly status reports for submission to the PM.

5.4.2 Weekly Status Reports

To facilitate timely review by project management, photocopies of logbook/notebook entries may be made for internal use.

In addition to those described herein, other summary reports may also be contractually required.

All TtNUS field forms can be found on the company's Intranet site at http://intranet.ttnus.com under Field Log Sheets.

6.0 LISTING OF FIELD FORMS ON THE TtNUS INTRANET SITE

- Boring Log
- Container Sample and Inspection Sheet
- Daily Activities Checklist
- Daily Activities Record
- Equipment Calibration Log
- Field Task Modification Request
- Field Analytical Log sheet - Geochemical Parameters
- Groundwater Level Measurement Sheet
- Groundwater Sample Log Sheet
- Hydraulic Conductivity Test Data Sheet
- Low Flow Purge Data Sheet
- Bedrock Monitoring Well Construction (Stick Up)
- Bedrock Monitoring Well Construction Flush Mount
- Bedrock Monitoring Well Construction Open Hole
- Confining Layer Monitoring Well Construction
- Monitoring Well Development Record

- Monitoring Well Materials Certificate of Conformance
- Overburden Monitoring Well Construction Flush Mount
- Overburden Monitoring Well Construction Stick Up
- Packer Test Report Form
- Pumping Test Data Sheet
- QA Sample Log Sheet
- Soil/Sediment Sample Log Sheet
- Surface Water Sample Log Sheet
- Test Pit Log
- Field Project Pre-Mobilization Checklist

ATTACHMENT A
TYPICAL SITE LOGBOOK ENTRY

START TIME: _____ DATE: _____

SITE LEADER: _____

PERSONNEL:

TtNUS	DRILLER	SITE VISITORS
_____	_____	_____
_____	_____	_____
_____	_____	_____

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenney and fire hoses were set up.
2. Drilling activities at well ____ resumes. Rig geologist was _____. See Geologist's Notebook, No. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4-inch stainless steel well installed. See Geologist's Notebook, No. 1, page 31, and well construction details for well _____.
3. Drilling rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well _____.
4. Well _____ drilled. Rig geologist was _____. See Geologist's Notebook, No. 2, page ____ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.
5. Well _____ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for 1 hour. At the end of the hour, water pumped from well was "sand free."
6. EPA remedial project manger arrives on site at 14:25 hours.
7. Large dump truck arrives at 14:45 and is steam-cleaned. Backhoe and dump truck set up over test pit _____.
8. Test pit _____ dug with cuttings placed in dump truck. Rig geologist was _____. See Geologist's Notebook, No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow groundwater table, filling in of test pit ___ resulted in a very soft and wet area. A mound was developed and the area roped off.
9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hours. Site activities terminated at 18:22 hours. All personnel off site, gate locked.

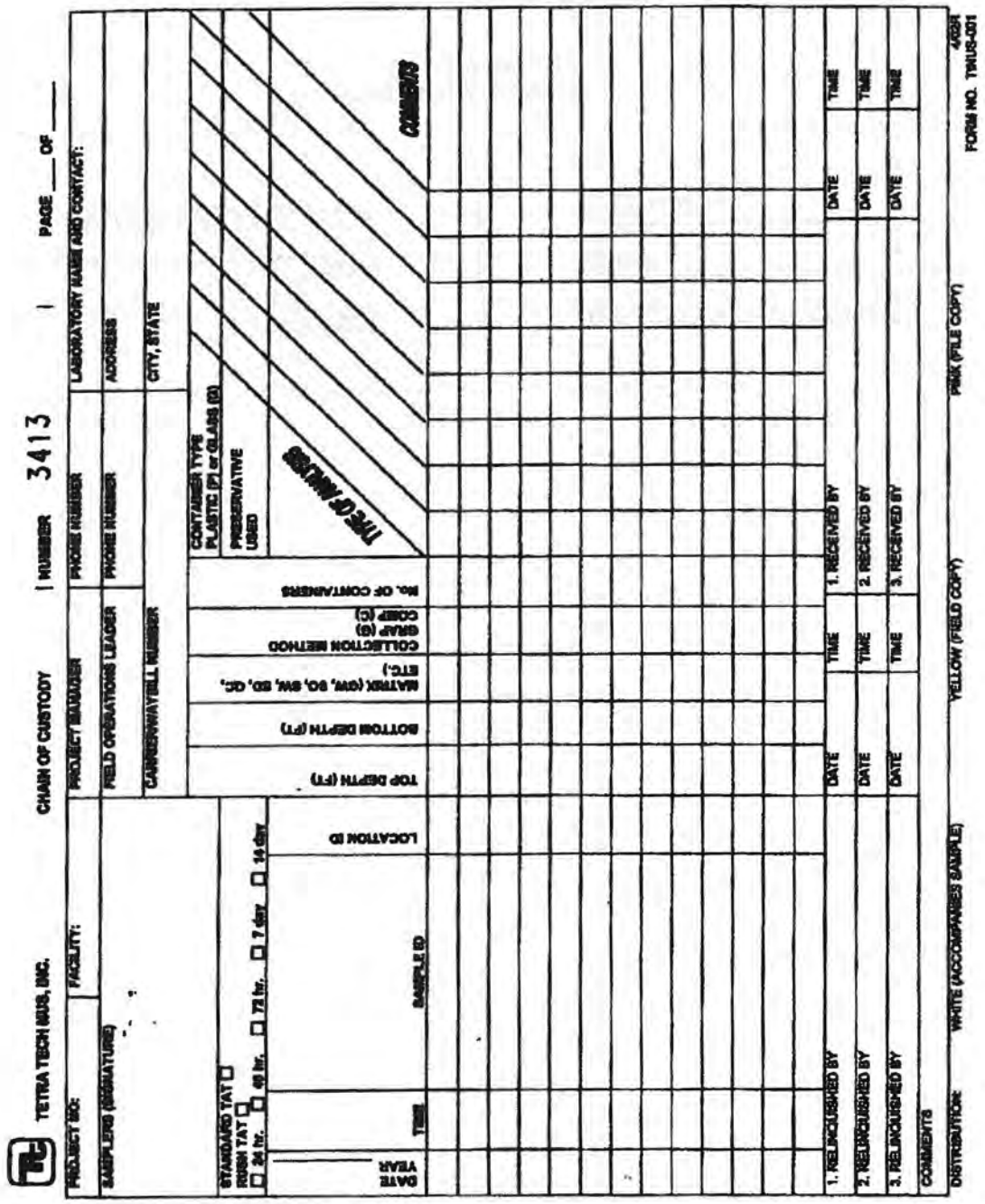
_____

Field Operations Leader

ATTACHMENT B
SAMPLE LABEL

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090	Project: Site: Location:	
Sample No:		Matrix:
Date:	Time:	Preserve:
Analysis:		
Sampled by:	Laboratory:	

ATTACHMENT C
CHAIN-OF-CUSTODY RECORD FORM



ATTACHMENT D
CHAIN-OF-CUSTODY SEAL

CUSTODY SEAL
Signature
Date

CUSTODY SEAL
Date
Signature

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		Effective Date 01/28/2009	Revision 6
		Applicability Tetra Tech NUS, Inc.	
		Prepared Earth Sciences Department	
Subject DECONTAMINATION OF FIELD EQUIPMENT		Approved Tom Johnston	

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ATTACHMENTS

1.0 PURPOSE

Decontamination is the process of removing and/or neutralizing site contaminants that have contacted and/or accumulated on equipment. The purpose of this Standard Operating Procedure (SOP) is to protect site personnel, the general public, and the environment while preserving or maintaining sample integrity. It is further intended through this procedure to describe the steps necessary for proper decontamination of drilling equipment, earth-moving equipment , chemical sampling equipment and field operation and analytical equipment.

2.0 SCOPE AND APPLICABILITY

This procedure applies to all equipment used to provide access to/acquire environmental samples that may have become contaminated through direct contact with contaminated media including air, water, and soil. This equipment includes drilling and heavy equipment and chemical sampling and field analytical equipment. Where technologically and economically feasible, single-use sealed disposable equipment will be employed to minimize the potential for cross-contamination. This SOP also provides general reference information on the control of contaminated materials.

Decontamination methods and equipment requirements may differ from one project to another. General equipment items are specified in Section 6.0, but project-specific equipment must be obtained to address the project-specific decontamination procedures presented in Section 7.0 and applicable subsections.

3.0 GLOSSARY

Alconox/Liquinox - A brand of phosphate-free laboratory-grade detergent.

Decontamination Solution - A solution selected/identified in the Health and Safety Plan or Project-Specific Quality Assurance Plan. The solution is selected and employed as directed by the project chemist/health and safety professional.

Deionized Water (DI) - Tap water that has been treated by passing through a standard deionizing resin column. This water may also pass through additional filtering media to attain various levels of analyte-free status. The DI water should meet College of American Pathologists (CAP) and National Committee for Clinical Laboratory Standards (NCCLS) specifications for reagent-grade Type I water.

Potable Water - Tap water from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute for tap water.

Pressure Washing - Process employing a high-pressure pump and nozzle configuration to create a high-pressure spray of potable water. High-pressure spray is employed to remove solids from equipment.

Solvent – A liquid in which solid chemicals or other liquids are dissolved. The solvent of choice is pesticide-grade isopropanol. Use of other solvents (methanol, acetone, or hexane) may be required for particular projects or for a particular purpose (e.g., removal of concentrated waste) and must be justified in the project planning documents. For example, it may be necessary to use hexane when analyzing for trace levels of pesticides, PCBs, or fuels. In addition, because many of these solvents are not miscible in water, the equipment should be air dried prior to use. Solvents should not be used on PVC equipment or well construction materials.

Steam Pressure Washing - A cleaning method employing a high-pressure spray of heated potable water to remove various organic/inorganic chemicals from equipment.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - Responsible for ensuring that all field activities are conducted in accordance with approved project plan(s) requirements.

Decontamination Personnel - Individuals assigned the task of decontamination. It is the responsibility of these individuals to understand the use and application of the decontamination process and solutions as well as the monitoring of that process to ensure that it is working properly. This is accomplished through visual evaluation, monitoring instrument scanning of decontaminated items, and/or through the collection of rinsate blanks to verify contaminant removal.

Field Operations Leader (FOL) - Responsible for the implementation of project-specific planning documents. This includes on-site verification that all field activities are performed in compliance with approved SOPs or as otherwise dictated by the approved project plan(s). The FOL is also responsible for the completion and accuracy of all field documentation.

Site Safety Officer (SSO) - Exercises shared responsibility with the FOL concerning decontamination effectiveness. All equipment arriving on site (as part of the equipment inspection), leaving the site, and moving between locations is required to go through a decontamination evaluation. This is accomplished through visual examination and/or instrument screening to determine the effectiveness of the decontamination process. Improper or incomplete decontamination is sufficient to restrict equipment from entering the site, exiting the site, or moving to a new location on the site until the objectives are successfully completed.

General personnel qualifications for decontamination activities include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.
- Familiarity with appropriate decontamination procedures.

5.0 HEALTH AND SAFETY

In addition to the health and safety issues and reminders specified in subsections of this SOP, the following considerations and requirements must be observed as SOPs for field equipment decontamination activities:

- If any solvents or hazardous chemicals (e.g., isopropyl alcohol) are to be used in equipment decontamination activities, the FOL must first obtain the manufacturer's/supplier's Material Safety Data Sheet (MSDS) and assure that it is reviewed by all users (prior to its use), added to the site Hazardous Chemical Inventory, and maintained on site as part of the project Hazard Communication Program.
- Review and observe specific health and safety requirements (e.g., personal protective equipment [PPE]) specified in the project-specific health and safety plan for this activity.

6.0 EQUIPMENT LIST

- Wood for decontamination pad construction, when applicable (see Section 7.1).

- Tools for constructing decontamination pad frame, when applicable (see Section 7.1).
- Visqueen sheeting or comparable material to cover decontamination pad frame, when applicable (see Section 7.1).
- Wash/drying racks for auger flights and drill/drive rods, when applicable (see Section 7.2).
- PPE as specified in the project health and safety plan.
- Soap and water for washing and rinsing.
- Deionized water for final rinsing.
- Solvents (e.g., pesticide-grade isopropanol) for rinsing (see applicable portions of Section 7.2).
- Tubs, buckets, etc. for containerizing rinse water (see applicable portions of Section 7.2).
- Sample bottles for collecting rinsate blanks (see Section 7.2).
- Calibrated photoionization detector (PID) or flame ionization detector (FID) to monitor decontaminated equipment for organic vapors generated through the existence of residual contamination or the presence of decontamination solvent remaining after the piece was rinsed.
- Aluminum foil or clear clean plastic bag for covering cleaned equipment (see applicable portions of Section 7.2).
- Paper towels or cloths for wiping.
- Brushes, scrapers, or other hand tools useful for removing solid materials from equipment.
- Clear plastic wrap for covering or wrapping large decontaminated equipment items (see Section 7.2.2).
- Drum-moving equipment for moving filled waste drums (optional) (see Section 7.3).
- Drum labels for waste drums (see Attachment A).

7.0 PROCEDURES

The process of decontamination is accomplished through the removal of contaminants, neutralization of contaminants, or isolation of contaminants. To accomplish this activity, preparation is required including site preparation, equipment selection, and evaluation of the decontamination requirements and processes. Site contaminant types, concentrations, and media types are primary drivers in the selection of the types of decontamination and where it will be conducted. For purposes of this SOP, discussion is limited to decontamination procedures for general environmental investigations.

Decontamination processes will be performed at the location(s) specified in project-specific planning documents. Typical decontamination locations include the following:

- Temporary decontamination pads/facilities
- Sample locations
- Centralized decontamination pad/facilities

- Combination of some or all of the above

The following discussion includes general considerations for the decontamination process. Specific construction and implementation procedures will be as specified in the project-specific planning documents and/or may be as dictated by site-specific conditions as long as the intent of the requirements in the planning documents is met. This intent is to contain any residual fluids and solids generated through the decontamination process.

7.1 Decontamination Pad Design/Construction Considerations

7.1.1 Temporary Decontamination Pads

Temporary decontamination pads may be constructed at satellite locations within the site area in support of temporary work areas. These structures are generally constructed to support the decontamination of heavy equipment such as drill rigs and earth-moving equipment but can be employed for smaller articles.

The purpose of the decontamination pad is to contain wash waters and potentially contaminated soil generated during decontamination procedures. Therefore, construction of these pads should take into account the following considerations:

- Site location – The decontamination site selected should be far enough from the work site to maximize decontamination effectiveness while minimizing travel distance. The location of the decontamination site shall be selected to provide, in the judgment of the FOL or FOL designee, compliance with as many of the following characteristics as practicable:

  - Well removed from pedestrian/vehicle thoroughfares.
  - Avoidance of areas where control/custody cannot be maintained.
  - Avoidance of areas where potential releases of contaminated media or decontamination fluids may be compounded through access to storm water transport systems, streams, or other potentially sensitive areas.
  - Avoidance of potentially contaminated areas.
  - Avoidance of areas too close to the ongoing operation, where cross-contamination may occur.

  The selected decontamination site should include the following, where possible:

  - Areas where potable water and electricity are provided.

> ***Safety Reminder***
>
> When utilizing electrical power sources, either hard-wired or portable-generated sources, ensure that:
>
> - All power is routed through a Ground Fault Circuit Interrupter (GFCI).
>
> - All power cords are in good condition (no physical damage), rated for the intended energy load, and designated for outdoor use.
>
> In situations where accomplishing these elements is not possible, it will be necessary to implement a site electrical grounding program.

- Areas where support activities such as removing decontamination waters soil and sediment are possible without entering an active exclusion zone.
- Areas that offer sufficient size to carry out the specific decontamination sequence.

- Decontamination pad (decon pad) – The decon pad shall be constructed to meet the following characteristics:
  - Size – The size of the pad should be sufficient to accept the equipment to be decontaminated as well as permitting free movement around the equipment by the personnel conducting the decontamination. The size should permit these movements utilizing pressure/steam washer wands and hoses and minimizing splash due to work in close quarters.
  - Slope – An adequate slope will be constructed to permit the collection of water and potentially contaminated soil within a trough or sump constructed at one end. The collection point for wash waters should be of adequate distance that the decontamination workers do not have to walk through the wash waters while completing their tasks. Because the pad will be sloped, place a light coating of sand over the plastic to minimize potential slips and falls. See the text about liners below.
  - Sidewalls – The sidewalls shall be at least 6 inches in height (or as high as possible if 6 inches is not achievable) to provide adequate containment for wash waters and soil. If splash represents a potential problem, splash guards should be constructed to control overspray. Sidewalls may be constructed of wood, inflatables, sand bags, etc. to permit containment. Splash guards are typically wood frames with Visqueen coverings to control overspray.
  - Liner – Depending on the types of equipment and decontamination method to be used, the liner should be of sufficient thickness to provide a puncture-resistant barrier between the decontamination operation and the unprotected environment. Care should be taken to examine the surface area prior to placing the liner to remove sharp articles (sticks, stones, debris) that could puncture the liner. Liners are intended to form an impermeable barrier. The thickness may vary from a minimum recommended thickness of 10 mil to 30 mil. The desired thickness may be achieved through layering materials of lighter construction. It should be noted that various materials (rubber, polyethylene sheeting) become slippery when wet. To minimize this potential hazard associated with a sloped liner, a light coating of sand shall be applied to provide traction as necessary.
  - Wash/drying racks – Auger flights, drill/drive rods, and similar equipment require racks positioned off of the ground to permit these articles to be washed, drained, and dried while secured from falling during this process.

For decontamination of direct-push technology (DPT) equipment, the pad may be as simple as a mortar tub containing buckets of soapy water for washing and an empty bucket to capture rinse waters. Decontamination may be conducted at the rear of the rig to permit rapid tool exchange.

- Maintenance – Maintain the decontamination area by:
  - Periodically clearing the work area of standing water, soil, and debris, and coiling hoses to aid in eliminating slip, trip, and fall hazards. In addition, these articles will reduce potential backsplash and cross-contamination.

- Regularly changing the decontamination fluids to ensure proper cleaning and prevent cross-contamination.

- PPE – Periodically evaluate the condition of, and maintain the decontamination equipment, including regular cleaning of face shields and safety glasses. This is critical to ensuring the safety of decontamination personnel and the integrity of the decontamination process, and it will ensure that equipment is functioning properly.

7.1.2 Decontamination Activities at Drill Rigs/DPT Units

During subsurface sampling activities including drilling and DPT activities, decontamination of drive rods, Macro Core Samplers, split spoons, etc. is typically conducted at an area adjacent to the operation. Decontamination is generally accomplished using a soap/water wash and rinse utilizing buckets and brushes. This area requires sufficient preparation to accomplish the decontamination objectives.

Buckets shall be placed within mortar tubs or similar secondary containment tubs to prevent splash and spills from reaching unprotected environmental media. Drying racks shall be employed as directed for temporary pads to permit parts to dry and be evaluated prior to use/reuse. Methodology regarding this activity is provided in Section 7.2.

7.1.3 Decontamination Activities at Remote Sample Locations

When sampling at remote locations, sampling equipment such as trowels and pumps/tubing should be evacuated of potentially contaminated media to the extent possible. This equipment should be wrapped in plastic for transport to the temporary/centralized decontamination location for final cleaning and disposition. Flushing and cleaning of single-use equipment such as disposable trowels, tubing, and surgeon's gloves may allow disposal of this equipment after visible soil and water remnants have been removed.

7.2 Equipment Decontamination Procedures

The following represents procedures to be employed for the decontamination of equipment that may have contacted and/or accumulated contamination through site investigation activities.

7.2.1 Monitoring Well Sampling Equipment

7.2.1.1 Groundwater sampling equipment – This includes pumps inserted into monitoring wells such as bladder pumps, Whale pumps, and Redi-Flo pumps and reusable bailers, etc.

1. Evacuate to the extent possible, any purge water within the pump/bailer.

2. Scrub using soap and water and/or steam clean the outside of the pump/bailer and, if applicable, the pump tubing.

3. Insert the pump and tubing/bailer into a clean container of soapy water. Pump/run a sufficient amount of soapy water through the pump/bailer to flush out any residual well water. After the pump is flushed, circulate soapy water through the pump to ensure that the internal components are thoroughly flushed.

4. Remove the pump and tubing/bailer from the container

5. Rinse external pump components using tap water.

6. Insert the pump and tubing/bailer into a clean container of tap water. Pump/run a sufficient amount of tap water through the pump/bailer to evacuate all of the soapy water (until clear).

> ***CAUTION***
> Do not rinse PE, PVC, and associated tubing with solvents –
> Use the procedures defined in the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 7 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

7. If groundwater contains or is suspected to contain oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment to be cleaned with pesticide-grade isopropanol.

8. Pass deionized water through the hose to flush out the tap water and solvent residue as applicable.

9. Drain residual deionized water to the extent possible.

10. Allow components of the equipment to air dry.

11. For bladder pumps, disassemble the pump and wash the internal components with soap and water, then rinse with tap water, isopropanol, and deionized water and allow to dry. After the parts are dry, conduct a visual inspection and a monitoring instrument scan to ensure that potential contaminants and all decontamination solvent have been removed. Collect a rinsate blank in accordance with the project-specific planning documents to ensure that the decontamination process is functioning as intended. The typical frequency of collection for rinsate blanks is 1 per 20 field samples. In addition, wipe samples or field tests such as UV light may be used.

12. Wrap pump/bailer in aluminum foil or a clear clean plastic bag for storage.

> ***SAFETY REMINDER***
> Remember when handling powered equipment to disconnect the power source and render the equipment to a zero energy state (both potential and kinetic) before opening valves, disconnecting lines, etc.

7.2.1.2 Electronic Water Level Indicators/Sounders/Tapes

During water level measurements, rinsing the extracted tape and probe with deionized water and wiping the surface of the extracted tape between locations is acceptable. However, periodic full decontamination should be conducted as follows:

1. Wash with soap and water
2. Rinse with tap water
3. Rinse with deionized water

> ***NOTE***
> In situations where oil, grease, free product, other hard to remove materials are encountered, probes and exposed tapes should be washed in hot soapy water. If probes or tapes cannot be satisfactorily decontaminated (they are still stained, discolored, etc.), they should be removed from service.

7.2.1.3 Miscellaneous Equipment

Miscellaneous equipment including analytical equipment (water quality testing equipment) shall be cleaned per manufacturers' instructions. This generally includes wiping the sensor housing and rinsing with tap and deionized water.

Coolers/shipping containers employed to ship samples are received from the laboratory in a variety of conditions including marginal to extremely poor. Coolers shall be evaluated prior to use for the following:

- Structural integrity – Coolers missing handles or having breaks in the outer housing should be removed and not used. Notify the laboratory that the risk of shipping samples in the cooler(s) provided is too great and request a replacement unit.

- Cleanliness – As per protocol, only volatile organic samples are accompanied by a trip blank. If a cooler's cleanliness is in question (visibly dirty/stained) or if there are noticeable odors, the cooler should be decontaminated prior to use as follows:

  1. Wash with soap and water
  2. Rinse with tap water
  3. Dry

If these measures fail to clean the cooler to an acceptable level, remove the unit from use as a shipping container and ask the cooler provider (e.g., the analytical laboratory) to provide a replacement unit.

7.2.2 Downhole Drilling Equipment

This includes any portion of the drill rig that is over the borehole, including auger flights, drill stems, rods, and associated tooling that would extend over the borehole. The following procedure is to be employed prior to initiating the drilling/sampling activity, then between locations:

> ***CAUTION***
> Exercise care when using scrapers to remove soil and debris from downhole drilling equipment. Inadvertent slips of scrapers have resulted in cuts, scrapes, and injured knuckles, so use scrapers carefully when removing soil from these items.

1. Remove loose soil using shovels, scrapers, etc.

2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment being decontaminated.

> ***CAUTION***
> In Step 3, do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

3. Rinse the equipment with tap water, where applicable (steam cleaning and pressure washing incorporate rinsing as part of the process).

4. If the equipment has directly or indirectly contacted contaminated sample media and is known or suspected of being contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse equipment with pesticide-grade isopropanol

5. To the extent possible, allow components to air dry.

6. If the decontaminated equipment is to be used immediately after decontamination, screen it with a calibrated photoionization detector (PID)/flame ionization detector (FID) to ensure that all contaminants and possible decontamination solvents (if they were used) have been adequately removed.

7. Wrap or cover equipment in clear plastic until it is time to be used.

SAFETY REMINDER

Even when equipment is disconnected from power sources, dangers such as the following may persist:

Falls - An auger flight standing on its end may fall and injure someone. Secure all loose articles to prevent heavy articles from falling onto people or equipment.

Burns - Steam cleaner water is heated to more than 212 °F and exhibits thermal energy that can cause burns. Prevent contact of skin with hot water or surfaces.

High water pressure - Pressure washer discharge can have 2,000 to 4,000 psi of water pressure. Water under this amount of pressure can rupture skin and other human tissues. Water at 4,000 psi exiting a 0° tip can be dangerous because of its relatively high cutting power. The exit velocity and cutting power of the water are reduced when exiting a 40° fan tip, but damage to soft tissues is still possible.

In general, follow the rules below to avoid injury, equipment damage, or incomplete decontamination:

1. Read the operating manual and follow the manufacturers' recommended safety practices before operating pressure washers and steam cleaners.

2. Never point the pressure washer or steam cleaner at another person or use to clean your boots or other parts of your body. Water lacerations and burns may appear to be minor at first but can be life threatening. Do not attempt to hold small parts in your hand while washing them with high-temperature or high-pressure water.

3. Always wear PPE as specified in the HASP such as:

   - Hard hat, safety glasses, splash shield, impermeable apron or splash suit, and hearing protection. Remember that excessive noise is a hazard when operating gas-powered engines and electrically driven pressure washers. PPE will be identified in your project specific planning documents.

4. Inspect each device before use. An inspection checklist will be provided in the project-specific planning documents. If it is a rented device, safety measures are typically provided by the vendor. In all cases, if you are not familiar with the operation of a pressure washer/steam cleaner, do not operate it until you obtain and thoroughly review operating instructions and recommended safety practices.

5. Do not modify equipment unless the manufacturer has approved the modifications.

7.2.3 Soil/Sediment Sampling Equipment

This section applies to soil sampling equipment including but not limited to hand augers, stainless steel trowels/spoons, bowls, dredges, scoops, split spoons, Macro Core samplers, etc.

1. Remove all loose soil from the equipment through manual means.
2. Through a combination of scrubbing using soap and water and/or steam cleaning or pressure washing, remove visible dirt/soil from the equipment.
3. Rinse the equipment with tap water.

> ***CAUTION***
> Do not rinse PE, PVC, and associated tubing with solvents. The appropriate procedures should be defined within the project-specific planning documents. If they are not defined, contact the FOL for guidance. The solvent rinse described in Step 4 may be omitted if groundwater does not contain oil, grease, PAHs, PCBs, or other hard to remove organic materials.

4. If the equipment is contaminated or suspected to be contaminated with oil, grease, PAHs, PCBs, or other hard to remove organic materials, rinse the equipment with pesticide-grade isopropanol.
5. Rinse the equipment with deionized water.
6. To the extent possible, allow components to air dry.
7. If the equipment is to be used immediately after decontamination, screen it with a calibrated PID/FID to ensure that all solvents (if they were used) and trace contaminants have been adequately removed.
8. After the equipment has dried, wrap it in aluminum foil for storage until use.

Dredges employed in sediment sampling are typically decontaminated as follows:

- Remove the sediment sample from the sampling device
- If sufficient associated surface water is available at the sampling site, place the dredge in the water and flush to remove visible sediment.
- Extract the dredge and wash it in soap and water per the project-specific planning documents.

> ***CAUTION***
> When handling dredges, the primary safety concern is trapping fingers or extremities in the larger dredge samplers within the jaws or pinch points of the mechanical jaws. Keep hands, fingers, and extremities away from these pinch and compression points. Either handle the device by the rope or preferably lock the jaws in place to control the potential for closing during maintenance and/or cleaning.

7.3 Contact Waste/Materials

During the course of field investigations, disposable/single-use equipment becomes contaminated. These items include tubing, trowels, PPE (gloves, overboots, splash suits, etc.), and broken sample containers.

With the exception of the broken glass, single-use articles should be cleaned (washed and rinsed) of visible materials and disposed as normal refuse. The exception to this rule is that extremely soiled materials that cannot be cleaned shall be containerized for disposal in accordance with applicable federal, state, and local regulations.

7.3.1 Investigation-Derived Wastes - Decontamination Wash Waters and Sediments

> ***NOTE***
> Requirements for waste storage may differ from one facility to the next. Facility-specific directions for waste storage areas will be provided in project-specific documents, or separate direction will be provided by the Project Manager.

1. Assume that all investigation-derived waste (IDW) generated from decontamination activities contains the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. Waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

2. Where possible, use filtering systems to extend the use of water within a closed system wash unit to recycle water and to reduce possible waste amounts.

> ***NOTE***
> Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility.

3. Label waste storage containers appropriately labeled (see Attachment A).

4. Ensure that the IDW storage area is configured to meet the following specifications to permit access to the containers and to conduct spill/leak monitoring, sampling, and extraction when the disposal route is determined:

   - Enclose areas accessible by the general public using construction fencing and signs.
   - Stored materials in 55-gallon drums on pallets with four (or fewer) drums per pallet.
   - Maintain the retaining bolt and label on the outside of storage containers where readily visible.
   - Provide at least 4 feet of room between each row of pallets to allow access to containers for sampling, drum removal, and spill response.
   - As directed in project-specific planning documents, maintain an IDW Inventory List and provide the list to the site Point of Contact at the termination of each shift.
   - Maintain spill response equipment at the IDW storage area in case it is required for immediate access.

- Where possible, use equipment for moving containers. Where not possible, obtain help to manipulate containers.

CAUTION

Each container of water can weigh up to 490 pounds. Each 55-gallon drum of wet soil can weigh more than 750 pounds. Fill drums and temporary containers to 80 percent capacity to minimize spill and handling difficulties. Use drum carts to move filled drums.

See safe lifting techniques provided in Section 4.4 of the Tetra Tech NUS, Inc. Health and Safety Guidance Manual.

When placing drums, keep your fingers out of pinch and smash points such as between the drums. In some cases such as well development and/or purge water, you can place the drums to be filled on the pallet and transport materials in smaller easier to handle containers.

7.4 Decontamination Evaluation

Upon decontamination of equipment, determine the effectiveness of the decontamination process in the following manner:

- Visual evaluation – A visual evaluation will be conducted to ensure the removal of particulate matter. This shall be done to ensure that the washing/rinsing process is working as intended.

- Instrument Screening – A properly calibrated PID/FID should be used to evaluate the presence of site contaminants and solvents used in the cleaning process. The air intake of the instrument shall be passed over the article to be evaluated. Avoid placing the instrument probe into residual waters. A PID/FID reading greater than the daily established background level requires a repeat of the decontamination process, followed by rescreening with the PID/FID. This sequence must be repeated until no instrument readings greater than the daily established background level are observed. It should be noted that the instrument scan is only viable if the contaminants are detectable within the instrument's capabilities.

NOTE

When required by project-specific planning documents, collection of rinsate blanks (see next step) shall be completed without exception unless approval to not collect these samples is obtained from the Project Manager.

- Collection of Rinsate Blanks – It is recommended that rinsate samples be collected to:

  - Evaluate the decontamination procedure representing different equipment applications (pumps versus drilling equipment) and different decontamination applications.

  - Single-use disposable equipment – The number of samples should represent different types of equipment as well as different lot numbers of single-use articles.

  - The collection and the frequency of collection of rinsate samples are as follows unless specified differently in the project-specific planning documents:

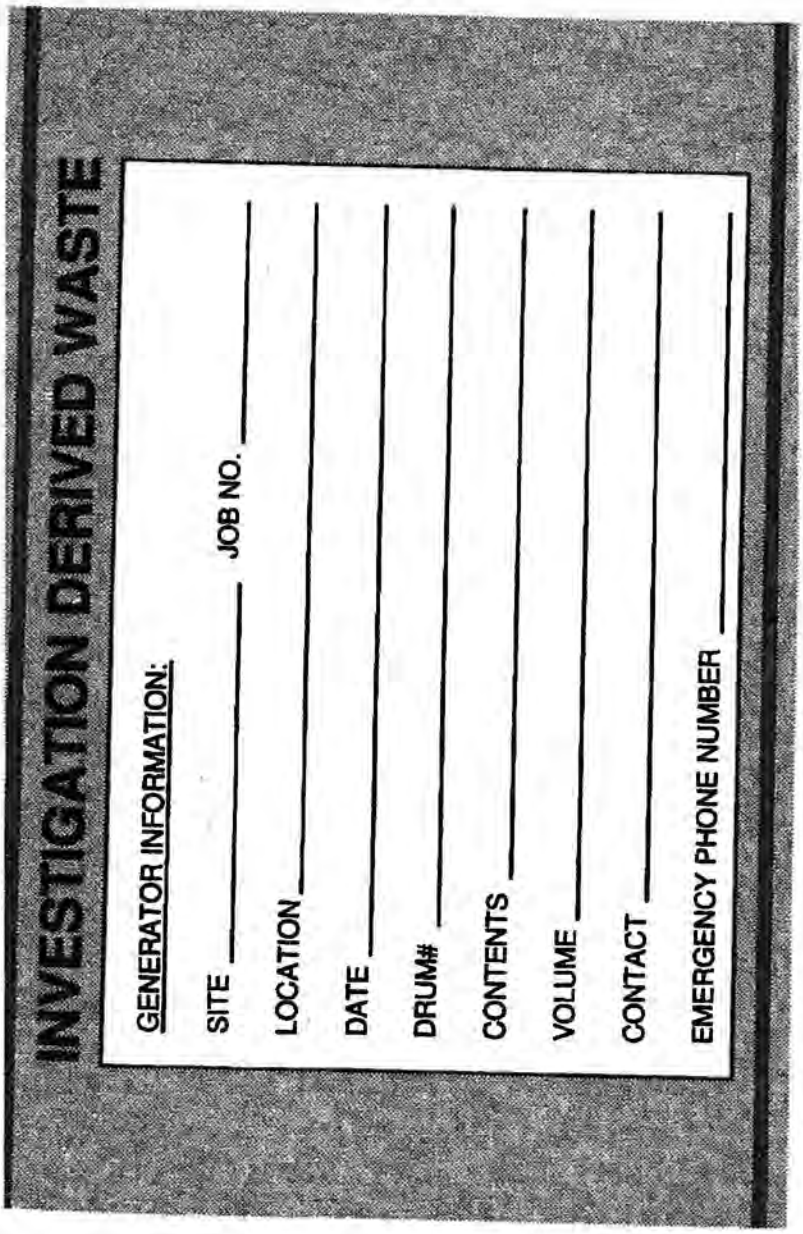
    - Per decontamination method
    - Per disposable article/batch number of disposable articles

NOTE

It is recommended that an initial rinsate sample be collected early in the project to ensure that the decontamination process is functioning properly and to avoid using a contaminated batch of single-use articles. It is recommended that a follow-up sample be collected later during the execution of the project to ensure that those conditions do not change.

Rinsate samples collection may be driven by types of and/or levels of contaminant. Difficult to remove contaminants, oils/greases, some PAHs/PCBs, etc. may also support the collection of additional rinsates due to the obvious challenges to the decontamination process. This is a field consideration to be determined by the FOL.

Attachment A
IDW Label



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		Effective Date 01/2012	Revision 8
		Applicability Tetra Tech, Inc.	
		Prepared Earth Sciences Department	
Subject GROUNDWATER SAMPLE ACQUISITION AND ONSITE WATER QUALITY TESTING		Approved J. Zimmerly	

TABLE OF CONTENTS

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the process to be used for purging groundwater monitoring wells prior to sampling, for collecting groundwater samples, and for measuring groundwater quality parameters.

2.0 SCOPE

This document provides information on proper sampling equipment, onsite water quality testing, safety measures to ensure the safety of the field technician(s), and techniques for groundwater sampling. All personnel are encouraged to review the information contained herein to facilitate planning of the field sampling effort. The techniques described shall be followed whenever applicable, noting that site-specific conditions or project-specific plans may require modifications to methodology.

3.0 GLOSSARY

Conductivity – Conductivity is a numerical expression of the ability of an aqueous solution to carry an electric current. This ability depends on the presence of ions and their total concentration, mobility, valence, and relative concentrations and on temperature. Conductivity is highly dependent on temperature and should be reported at a particular temperature, i.e., 20.2 microSiemens per centimeter (mS/cm) at 14°C.

Dissolved Oxygen (DO) – DO levels in natural and wastewater depend on the physical, chemical, and biochemical activities in the water sample.

Groundwater Sample – A quantity of water removed from the ground, usually via a monitoring well that may or may not be lined with a well casing.

Oxidation-Reduction Potential (ORP) - A measure of the activity ratio of oxidizing and reducing species as determined by the electromotive force developed by a noble metal electrode immersed in water, as referenced against a reference electrode. A reference electrode commonly used in the field is the silver/silver chloride electrode, which has a voltage offset of about 210 mV from the standard hydrogen electrode (SHE). To convert field ORP measurements to equivalent SHE values, approximately 210 mV must be added to the ORP values obtained using the silver/silver chloride electrode. The actual offset depends on the concentration of the potassium chloride (KCl) in the field reference electrode and the temperature. Offsets typically range from 199 (saturated KCl) to 205 (3.5 Molar KCl) to 222 mV (1 Molar KCl) at 25°C and are greater at lower temperatures.

pH - The negative logarithm (base 10) of the hydrogen ion activity. The hydrogen ion activity is related to the hydrogen ion concentration, and, in a relatively weak solution, the two are nearly equal. Thus, for all practical purposes, pH is a measure of the hydrogen ion concentration.

pH Paper - Indicator paper that turns different colors depending on the pH of the solution to which it is exposed. Comparison with color standards supplied by the manufacturer will then give an indication of the solution's pH.

Representativeness – A qualitative description of the degree to which an individual sample accurately reflects population characteristics or parameter variations at a sampling point. It is therefore an important characteristic not only of assessment and quantification of environmental threats posed by the site, but also for providing information for engineering design and construction. Proper sample location selection and proper sample collection methods are important to ensure that a truly representative sample has been collected.

Salinity – The measurement of dissolved salts in a given mass of solution. Note: most field meters determined salinity automatically from conductivity and temperature. The value will be displayed in either parts per thousand (ppt) or percent (e.g., 35 ppt equals 3.5 percent). The parts per thousand symbol ($^0/_{00}$) is not the same as the percent symbol (%).

Turbidity – Turbidity in water is caused by suspended matter such as clay, silt, and fine organic and inorganic matter. Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample.

4.0 RESPONSIBILITIES AND PERSONNEL QUALIFICATIONS

Project Manager - The Project Manager is responsible for determining the sampling objectives, initial sampling locations, and field procedures used in the collection of groundwater samples. Additionally, in consultation with other project personnel (geologist, hydrogeologist, etc.), the Project Manager identifies sampling locations.

Site Safety Officer (SSO) - The SSO (or a qualified designee) is responsible for providing the technical support necessary to implement the project Health and Safety Plan (HASP). This includes but is not be limited to performing air quality monitoring during sampling, boring and excavation activities, and ensuring that workers and offsite (downwind) individuals are not exposed to hazardous levels of airborne contaminants. The SSO or SSO designee may also be required to advise the FOL on other safety-related matters regarding sampling, such as mitigative measures to address potential hazards from hazardous objects or conditions.

Project Geologist/Sampler - The project geologist/sampler is responsible for the proper acquisition of samples in accordance with this SOP or other project-specific documents. In addition, this individual is responsible for the completion of all required paperwork (e.g., sample log sheets, field notebook, boring logs, container labels, custody seals, and chain-of-custody forms) associated with the collection of those samples.

Project Hydrogeologist – This individual is responsible for selecting and detailing the specific groundwater sampling techniques, onsite water quality testing (type, frequency, and location), equipment to be used, and providing detailed input in this regard to the project planning documents. The project hydrogeologist is also responsible for properly briefing and overseeing the performance of site sampling personnel.

Field Operations Leader (FOL) – This individual is primarily responsible for the execution of the planning document containing the Sampling and Analysis Plan (SAP). This is accomplished through management of a field sampling team for the proper acquisition of samples. He or she is responsible for the supervision of onsite analyses; ensuring proper instrument calibration, care, and maintenance; sample collection and handling; the completion and accuracy of all field documentation; and making sure that custody of all samples obtained is maintained according to proper procedures. When appropriate and as directed by the FOL, such responsibilities may be performed by other qualified personnel (e.g., field technicians) where credentials and time permit. The FOL is ultimately responsible for adherence to Occupational Safety and Health Administration (OSHA) regulations during these operations through self acquisition or through the management of a field team of samplers.
General personnel qualifications for groundwater sample collection and onsite water quality testing include the following:

- Occupational Safety and Health Administration (OSHA) 40-hour and applicable refresher training.
- Capability of performing field work under the expected physical and environmental (i.e., weather) conditions.

- Familiarity with appropriate procedures for sample documentation, handling, packaging, and shipping.

5.0 HEALTH AND SAFETY

Specific safety and health precautions are identified throughout this SOP. In addition to those precautions, the following general hazards may be incurred during sampling activities:

- Knee injuries from kneeling on hard surfaces
- Slips, trips, and falls
- Cuts and lacerations
- Traffic hazards associated with sampling in parking areas and roadways and along highways.

Methods of avoiding these hazards are provided below.

Knee injuries – Many monitoring wells are installed as flush mounts. Personnel are required to kneel to open these wells and to take groundwater level measurements, etc. This could result in knee injuries from kneeling on stones/foreign objects and general damage due to stress on the joints. To combat this hazard:

- Clear any foreign objects from the work area.
- Wear hard-sided knee pads.

Slips, Trips, and Falls – These hazards exist while traversing varying terrains carrying equipment to sample wells. To minimize these hazards:

- Pre-survey well locations. Eliminate, barricade, or otherwise mark physical hazards leading to the locations.
- Carry small loads that do not restrict the field of vision.
- Travel the safest and clearest route (not necessarily the shortest).

Cuts and Lacerations – To prevent cuts and lacerations associated with groundwater sampling, the following provisions are required:

- Always cut away from yourself and others when cutting tubing or rope. This will prevent injury to yourself and others if the knife slips.
- Do not place items to be cut in your hand or on your knee.
- Change blades as necessary to maintain a sharp cutting edge. Many accidents result from struggling with dull cutting attachments.
- Whenever practical, wear cut-resistant gloves (e.g., leather or heavy cotton work gloves) at least on the hand not using the knife.
- Keep cutting surfaces clean and smooth.

- Secure items to be cut -- do not hold them against the opposing hand, a leg, or other body part.
- When transporting glassware, keep it in a hard-sided container such as a cooler so that if there is a fall, you will be less likely to get cut by broken glass.
- DO NOT throw broken glass or glass ampoules into garbage bags. Place broken glass and glass ampoules in hard-sided containers such as a cardboard box or directly into a dumpster. DO NOT reach into garbage bags to retrieve any item accidentally thrown away. Empty the contents onto a flat surface to avoid punctures and lacerations from reaching where you cannot see.

Vehicular and Foot Traffic Hazards – When sampling along the roadway or near traffic patterns, follow the following precautions:

- Motorists may be distracted by onsite activities – ASSUME THEY DO NOT SEE YOU OR MEMBERS OF YOUR FIELD CREW.
- DO NOT place obstructions (such as vehicles) along the sides of the road that may cause site personnel to move into the flow of traffic to avoid your activities or equipment or that will create a blind spot.
- **Provide a required free space of travel**. Maintain at least 6 feet of space between you and moving traffic. Where this is not possible, use flaggers and/or signs to warn oncoming traffic of activities near or within the travel lanes.
- Face Traffic. Whenever feasible, if you must move within the 6 feet of the required free space or into traffic, attempt to face moving traffic at all times. Always leave yourself an escape route.
- Wear high-visibility vests to increase visual recognition by motorists.
- Do not rely on the vehicle operator's visibility, judgment, or ability. Make eye contact with the driver. Carefully and deliberately use hand signals so they will not startle or confuse motorists or be mistaken for a flagger's direction before moving into traffic.
- Your movements may startle a motorist and cause an accident, so move deliberately. Do not make sudden movements that might confuse a motorist.

6.0 PROCEDURES

6.1 General

For information derived from a groundwater sample to be useful and accurate, the sample must be representative of the particular zone being sampled. The physical, chemical, and bacteriological integrity of the sample must be maintained from the time of sampling to the time of analysis to keep any changes in water quality parameters to a minimum.

CAUTION

A closed well may generate and accumulate gases due to biological degradation, evolution of volatile chemicals from groundwater into the air, or other chemical actions. These gases may also be artificially generated, such as in the case of air sparging or

extraction wells, which may take several days to depressurize. See Section 6.6.2 for safety measures to be employed to protect sampling personnel.

Methods for withdrawing samples from completed wells include the use of pumps, compressed air or nitrogen, bailers, and various types of samplers. The primary considerations in obtaining a representative sample of groundwater are to avoid collection of stagnant (standing) water in the well and to avoid physical or chemical alteration of the water sample due to external influences of the sampling technique(s). In a non-pumping well, there will be little or no vertical mixing of water in the well pipe or casing, and stratification will occur. The well water in the screened section will mix with groundwater due to normal flow patterns, but the well water above the screened section will remain isolated and become stagnant. Concentration gradients resulting from mixing and dispersion processes, layers of variable geologic permeability, and the presence of separate-phase product (e.g., floating hydrocarbons) may cause stratification. Excessive pumping or improper sampling methods can dilute or increase contaminant concentrations in the collected sample compared to what is representative of the integrated water column as it naturally occurs at that point, resulting in the collection of a non-representative sample. To safeguard against collecting non-representative samples, the following approach shall be followed prior to sample acquisition:

CAUTION

Mechanical agitation of well water may cause off-gas generation of volatile contaminants, creating an inhalation exposure to the sampler(s). Where avoiding an inhalation exposure is not possible and mechanical agitation is possible, pump into closed-top containers to control potential air emissions.

1. If possible, position yourself (and the sampling equipment) upwind of the well head.

2. Purge the monitoring well to be sampled prior to obtaining any samples from it. Evacuation of three to five well volumes is recommended prior to sampling, unless low-flow purging and sampling methods are utilized as described in Section 6.7 (Consult the site-specific SAP for exact purging parameters). In a high-yielding groundwater formation and where there is no stagnant water in the well above the screened section, extensive evacuation prior to sample withdrawal is not as critical as it is in a low-yielding well or in wells containing stagnant water.

3. For wells with low yields that are purged dry during sampling, evacuate the well and allow it to recover to 75 percent of full capacity prior to sample acquisition. If the recovery rate is fairly rapid (generally 300 mL per minute or greater), attempt to continue evacuation until the number of well volumes specified in the SAP is achieved. If this cannot be accomplished, allow recovery to 75 percent of capacity and begin sampling.

CAUTION

For moderate to high-yielding monitoring wells, an evacuation rate that does not cause excessive turbulence in the well should be selected. There is no absolute safeguard against contaminating the sample with stagnant water; hence, special techniques are required for purging to minimize the potential for sample contamination (see below).

4. For moderate to high-yielding monitoring wells, use one of the following purge techniques:

   - Place a submersible pump or the intake line of a surface pump or bailer just below the water surface when removing the stagnant water.

- While purging and as the water level decreases, lower the pump or intake line as the water level drops in the well. Three to five volumes of water shall be removed to provide reasonable assurance that all stagnant water has been evacuated. After this is accomplished, a bailer or other approved device may be used to collect the sample for analysis.

- Unless otherwise directed, place the intake line of the sampling pump (or the submersible pump itself) near the center of the screened section, and pump approximately one casing volume of water from the well at a low purge rate equal to the well's recovery rate (low-flow sampling).

6.2 Sampling, Monitoring, and Evacuation Equipment

Sample containers shall conform to the guidelines in SOP SA-6.1.

The following equipment shall be on hand when sampling groundwater wells (reference SOPs SA-6.1 and SA-7.1):

- Sample packaging and shipping equipment – Coolers for sample shipping and cooling, chemical preservatives, appropriate sampling containers and filler materials, ice, labels, and chain-of-custody documents.

- Field tools and instrumentation

  - Multi-parameter water quality meter with an in-line sample chamber capable of measuring ORP, pH, temperature, DO, specific conductance, turbidity, and salinity, or individual meters (as applicable)

  - pH Paper

  - Camera and film (if appropriate)

  - Appropriate keys (for locked wells)

  - Water level indicator and/or oil-water interface probe if separate-phase product is expected

- Pumps

  - Shallow-well pumps: Centrifugal, bladder, suction, or peristaltic pumps with drop lines and air-lift apparatus (compressor and tubing) where applicable.

  - Deep-well pumps: Submersible pump and electrical power-generating unit, or bladder pumps where applicable.

- Other sampling equipment – Bailers, graduated cylinder, stopwatch, and inert line with tripod-pulley assembly (if necessary).

- Pails – Plastic, graduated.

- Clean paper or cotton towels for cleaning equipment.

- Buckets with lids for collecting purge water.

- Decontamination solutions – Deionized water, potable water, phosphate-free laboratory-grade detergent, and analytical-grade solvent (e.g., pesticide-grade isopropanol), as required.

Ideally, sample withdrawal equipment shall be completely inert, economical, easily cleaned, cleaned prior to use, reusable, able to operate at remote sites in the absence of power sources, and capable of delivering variable rates for well purging and sample collection.

6.3 Calculations of Well Volume

To ensure that the proper volume of water has been removed from the well prior to sampling, it is first necessary to know the volume of standing water in the well pipe (including well screen where applicable). This volume can be easily calculated by the following method. Calculations shall be entered in the site logbook or field notebook or on a sample log sheet form or equivalent electronic form(s) (see SOP SA-6.3):

1. Obtain all available information on well construction (location, casing, screen, etc.).
2. Determine well or inner casing diameter.
3. Measure and record static water level (depth below ground level or top of casing reference point).
4. Determine depth of well by sounding using a clean, decontaminated, weighted tape measure or water level indicator.
5. Calculate number of linear feet of static water (total depth or length of well pipe minus the depth to static water level).
6. Calculate one static well volume in gallons $V = (0.163)(T)(r^2)$

   where: V = Static volume of well in gallons.
   T = Linear feet of water in the well.
   r = Inside radius of well casing in inches.
   0.163 = Conversion factor (compensates for conversion of casing radius from inches to feet and cubic feet to gallons and pi.

7. Per evacuation volumes discussed above, determine the minimum amount to be evacuated before sampling.

Measuring devices may become contaminated when gathering the above information if they are submerged in contaminated water. Decontamination of the tape or water level indicator must be conducted between measurements in different wells as follows:

1. Saturate a paper towel or clean cotton towel with deionized water.
2. As the measuring device is extracted, wipe the tape, changing the cleaning surface frequently.
3. After it is extracted, rinse the probe or tape using a spray bottle of deionized water over a bucket or similar collection container.

Based on the contaminant (oily, etc), it may be necessary to use a soap and water wash and rinse to remove contaminants. Isopropanol can be used on the probe/tape. However, it is recommended that the use of solvents on the tape be minimized because they could degrade the protective covering or possibly

remove the scale designations. If isopropanol (or some other solvent) is used, assure that the manufacturer/supplier Material Safety Data Sheet (MSDS) is obtained, kept on site at a readily available location with other MSDSs, and reviewed by personnel prior to the first usage of the solvent. Also, add the substance to the site-specific Hazardous Chemical Inventory list (see Section 5 of the TtNUS Health and Safety Guidance Manual [HSGM], Hazard Communication Program and OSHA Standard 29 CFR 1910.1200).

6.4 Evacuation of Static Water – Purging

6.4.1 General

The amount to be purged from each well will be determined prior to sample collection. This amount will depend on the intent of the monitoring program and the hydrogeologic conditions. Programs to determine overall quality of water resources may require long pumping periods to obtain a sample that is representative of a large volume of the aquifer. The pumped volume may be specified prior to sampling so that the sample can be a composite of a known volume of the aquifer. Alternately, the well can be pumped until parameters such as temperature, specific conductance, pH, and turbidity (as applicable) have stabilized. Onsite measurements of these parameters shall be recorded in the site logbook or field notebook or on standardized data sheets or an equivalent electronic form(s).

6.4.2 Evacuation Devices

The following discussion is limited to those devices commonly used at hazardous waste sites. Attachment A provides guidance on the proper evacuation device to use for given sampling situations. All of these techniques involve equipment that is portable and readily available.

Bailers

Bailers are the simplest evacuation devices used and have many advantages. They generally consist of a length of tubing equipped with a base plate and ball check-valve at the bottom. Bailers are comprised of stainless steel and plastic. They come in a variety of sizes, but the two most often used are 2 inches and 4 inches in diameter. An inert non-absorbent line such as polyethylene rope is used to lower and then raise the bailer to retrieve the sample. As the bailer is lowered into the water column, the ball is pushed up allowing the tube to be filled. When the bailer is pulled upward, the ball seats in the base plate preventing water from escaping.

Advantages of bailers include the following:

- There are few limitations on size and materials used.
- No external power source is needed.
- Bailers are inexpensive and can be dedicated and hung in a well to reduce the chances of cross-contamination.
- Bailers are relatively easy to decontaminate.

Limitations on the use of bailers include the following:

- It is time consuming to remove stagnant water using a bailer.

- Splashing the bailer into the water or transfer of sample may cause aeration.
- The use of a bailer does not permit constant in-line monitoring of groundwater parameters.
- Use of bailers is physically demanding, especially in warm temperatures at personal protection equipment (PPE) levels above Level D.

Safety concerns using a bailer include the following:

- Muscle stress and strain, especially when using 4-inch bailers and when pulling from excessively deep wells.
- Entanglement, possible hand/finger injuries, and rope burns during a sudden release of the bailer back down the well.
- Direct contact with contaminants of concern and sample preservatives when discharging the bailer contents because there is not a high level of control during a direct pour, and splashing and indirect contact with contaminants/preservatives could occur.

Control measures for these hazards are provided in Section 6.6.2.

Suction Pumps

There are many different types of inexpensive suction pumps including centrifugal, diaphragm, and peristaltic pumps. Centrifugal and diaphragm pumps can be used for well evacuation at a fast pumping rate and for sampling at a low pumping rate. The peristaltic pump is a low-volume pump that uses rollers to squeeze flexible tubing to create suction. This tubing can be dedicated to a well to prevent cross-contamination from well to well. Suction pumps are all portable, inexpensive, and readily available. However, because they are based on suction, their use is restricted to areas with water levels within 20 to 25 feet of the ground surface. A significant limitation is that the vacuum created by these pumps can cause loss of dissolved gases and volatile organics. Another limitation of these pumps is that they require a secondary energy source to drive them. Electrically driven pumps may require portable generators as energy sources. Air diaphragm pumps require air compressors and/or compressed gas cylinders to drive them. The advantage of the peristaltic pump is that it will operate from a portable battery source. Safety measures associated with these pumps are provided below.

Air-Lift and Gas-Lift Samplers

This group of pump samplers uses gas pressure either in the annulus of the well or in a venturi to force groundwater up a sampling tube. These pumps are also relatively inexpensive. Air- or gas-lift samplers are more suitable for well development than for sampling because the samples may be aerated as a result of pump action. Aeration can cause pH changes and subsequent trace metal precipitation or loss of volatile organics.

Submersible Pumps

Submersible pumps take in water and push the sample up a sample tube to the surface. The power sources for these samplers may be compressed gas or electricity. Operation principles vary, and displacement of the sample can be by an inflatable bladder, sliding piston, gas bubble, or impeller. Pumps are available for 2-inch-diameter wells and larger. These pumps can lift water from considerable depths (several hundred feet).

Limitations of this class of pumps include the following:

- They may have low delivery rates.
- Many models are expensive.
- Compressed gas or electric power is needed.
- Sediment in water may cause clogging of the valves or eroding of the impellers with some of these pumps.
- Decontamination of internal components can be difficult and time consuming.

Compressed Gases

Safety concerns using compressed gases as an energy source in these pumps are numerous. The nitrogen gas or compressed air is provided in a compressed gas cylinder at a pressure of approximately 2,000 psi. If damaged, these cylinders can become dangerous projectiles. Additionally, a sudden release of a cylinder's contents can involve considerable force that could cause significant damage to the eyes and/or skin. Protective measures include the following:

- Always wear safety impact glasses when handling compressed gases.
- Always administer compressed gases through an appropriate pressure-reducing regulator.
- When clearing the cylinder connection port, open the cylinder valve only enough to clear foreign debris. During this process, always position the cylinder valve so that it faces away from you and others.
- If the cylinder is designed to accept a valve protection cap, always keep that protection cap in place, except the cylinder is connected for use.
- When using the cylinder, lay the cylinder on its side to avoid the potential of it falling and knocking the valve off (and becoming a missile).
- DO NOT use the compressed nitrogen or air to clean clothing or to spray off the skin. Small cuts in the protective layer of the skin may permit the gas to enter into the bloodstream, presenting the potential danger of an embolism.

See the project-specific HASP for additional direction concerning cylinder safe handling procedures pertaining to the safe handling, transportation, and storage of compressed gas cylinders.

Electrical Shock

Even in situations where portable batteries are used, the potential for electrical shock exists. This potential risk is increased in groundwater sampling activities because of the presence of groundwater near the batteries. This potential is also increased in (prohibited) situations where jury-rigging of electrical connections is performed. Other potential hazards occur when field samplers open the hood of a running car to access the battery as a power source. To control these hazards:

- If you are unfamiliar with electrical devices, do not experiment, get help, and get the proper equipment necessary to power your device.
- Use the proper portable power inverters for cigarette lighter connections to minimize the need to access the battery under the hood of your vehicle.
- Use of electrical generators may pose a number of hazards including noise, those associated with fueling, and indirect sample influence.

To minimize or eliminate electrical generator hazards:

- Inspect the generator before use. Ensure that the generator and any extension cords are rated for the intended operation and have a Ground Fault Circuit Interrupter (GFCI) in line to control potential electrical shock.
- Fuel the generator before purging and sampling to avoid loss of power during sampling.
- Fuel engines only when they are turned OFF and have cooled sufficiently to prevent a fire hazard.
- Place the generator and any fuel source at least 50 feet from the well to be sampled to avoid indirect influence to the sample from fuel vapors or emission gases.

Lifting Hazards

This hazard may be experienced when moving containers of purge water, equipment, cylinders, etc. To control these potential hazards:

- Do not fill purge buckets to more than 80 percent of their capacity.
- Obtain a gas cylinder of sufficient size to complete the designated task but not too large to handle. K-size cylinders weigh approximately 135 pounds and are difficult to handle. M-size cylinders weigh approximately 50 pounds and are easier to handle and move.
- When necessary, get help lifting and moving gas cylinders and other heavy objects. Minimize twisting and turning while lifting. If it is necessary to move these cylinders or generators over significant distance, use mechanical means (carts, etc.).
- Use proper lifting techniques as described in Section 4.4 of the HSGM.

6.5 Onsite Water Quality Testing

This section describes the procedures and equipment required to measure the following parameters of an aqueous sample in the field:

- pH
- Specific conductance
- Temperature
- DO
- ORP
- Turbidity
- Salinity

This section is applicable for use in an onsite groundwater quality monitoring program to be conducted at a hazardous or nonhazardous waste site. The procedures and equipment described are applicable to groundwater samples and are not, in general, subject to solution interferences from color, turbidity, or colloidal material or other suspended matter.

This section provides general information for measuring the parameters listed above with instruments and techniques in common use. Because instruments from different manufacturers may vary, review of the manufacturer's literature pertaining to the use of a specific instrument is required before use. Most meters used to measure field parameters require calibration on a daily basis. Refer to SOP SA-6.3 for an example equipment calibration log.

6.5.1 Measurement of pH

6.5.1.1 General

Measurement of pH is one of the most important and frequently used tests in water chemistry. Practically every phase of water supply and wastewater treatment such as acid-base neutralization, water softening, and corrosion control is pH dependent. Likewise, the pH of leachate can be correlated with other chemical analyses to determine the probable source of contamination. It is therefore important that reasonably accurate pH measurements be taken and recorded on the groundwater sample log sheet (Attachment B) or equivalent electronic form.

Two methods are given for pH measurement: the pH meter and pH indicator paper. Indicator paper is used when only an approximation of the pH is required or when pH meter readings need to be verified, and the pH meter is used when a more accurate measurement is needed. The response of a pH meter can be affected by high levels of colloidal or suspended solids, but the effect is generally of little significance. Consequently, specific methods to overcome this interference are not described. The response of pH paper is unaffected by solution interferences from color, turbidity, or colloidal or suspended materials unless extremely high levels capable of coating or masking the paper are encountered. In such cases, use of a pH meter is recommended.

6.5.1.2 Principles of Equipment Operation

Use of pH papers for pH measurement relies on a chemical reaction caused by the acidity or alkalinity of the solution created by the addition of the water sample reacting with the indicator compound on the paper. Various types of pH papers are available, including litmus (for general acidity or alkalinity determination) and specific, or narrower range, pH range paper.

Use of a pH meter relies on the same principle as other ion-specific electrodes. Measurement relies on establishment of a potential difference across a glass or other type of membrane in response to (in this instance, hydrogen) ion activity (which is usually similar to concentration) across that membrane. The membrane is conductive to ionic species and, in combination with a standard or reference electrode, a potential difference proportional to the ion concentration is generated and measured.

6.5.1.3 Equipment

The following equipment is to be used for obtaining pH measurements:

- A stand-alone portable pH meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).

- Combination electrode with polymer body to fit the above meter. Alternately, a pH electrode and a reference electrode can be used if the pH meter is equipped with suitable electrode inputs.
- Buffer solutions, as specified by the manufacturer. If the buffer solutions are considered hazardous per 29 Code of Federal Regulations (CFR) 1910.1200 (Hazard Communication) or the volumes used are greater than consumer commodity levels, the SSO shall obtain MSDSs from the manufacturer for the specific buffer solutions (see Section 4 of the HSGM regarding the Hazard Communication Program)
- pH indicator paper to cover the pH range 2 through 12.
- Manufacturer's operation manual. All personnel must be familiar with the equipment operation to ensure that the integrity of samples is preserved and that the equipment is operated safely.

6.5.1.4 Measurement Techniques for Field Determination of pH

pH Meter

The following procedure shall be used for measuring pH with a pH meter (meter standardization is according to manufacturer's instructions):

1. Inspect the instrument and batteries prior to initiation of the field effort.
2. Check the integrity of the buffer solutions used for field calibration. Buffer solutions need to be changed often as a result of degradation upon exposure to the atmosphere.
3. If applicable, make sure all electrolyte solutions within the electrode(s) are at their proper levels and that no air bubbles are present within the electrode(s).
4. Calibrate the meter and electrode(s) on a daily use basis (or as recommended by manufacturer) following manufacturer's instructions. Record calibration data on a water quality meter calibration log sheet (Attachment C) or equivalent electronic form.
5. Immerse the electrode(s) in the sample. Stabilization may take several seconds to minutes. If the pH continues to drift, the sample temperature may not be stable, a physical reaction (e.g., degassing) may be taking place in the sample, or the meter or electrode may be malfunctioning. The failure of the measurements to stabilize must be clearly noted in the logbook or equivalent electronic form.
6. Read and record the pH of the sample. pH shall be recorded to the nearest 0.01 pH standard unit. Also record the sample temperature (unless otherwise specified in the SAP, record temperatures to the nearest whole degree Fahrenheit or 0.5 degree Celsius).
7. Rinse the electrode(s) with deionized water.
8. Store the electrode(s) in an accordance with manufacturer's instructions when not in use.

Any visual observation of conditions that may interfere with pH measurement, such as oily materials or turbidity, shall be noted and avoided as much as possible.

pH Paper

Use of pH paper is very simple and requires no sample preparation, standardization, etc. pH paper is available in several ranges, including wide-range (indicating approximately pH 1 to 12), mid-range (approximately pH 0 to 6, 6 to 9, 8 to 14) and narrow-range (many available, with ranges as narrow as 1.5 pH units). The appropriate range of pH paper shall be selected. If the pH is unknown the investigation shall start with wide-range paper and proceed with successively narrower range paper until the sample pH is determined. To measure the pH with pH paper:

1. Collect a small portion of sample into a clean container.
2. Dip the pH paper into this small portion of sample.
3. Compare the color of the paper to the color chart that is provided with the pH paper and read the corresponding pH from the chart.
4. Record the pH value from the chart on the sampling log sheet.
5. Discard the used pH paper as trash.
6. Discard the small volume of sample that was used for the pH measurement with the other investigative derived waste.

6.5.2 Measurement of Specific Conductance

6.5.2.1 General

Conductance provides a measure of dissolved ionic species in water and can be used to identify the direction and extent of migration of contaminants in groundwater or surface water. It can also be used as a measure of subsurface biodegradation or to indicate alternate sources of groundwater contamination.

Conductivity is a numerical expression of the ability of a water sample to carry an electric current. This value depends on the total concentration of ionized substances dissolved in the water and the temperature at which the measurement is made. The mobility of each of the various dissolved ions, their valences, and their actual and relative concentrations affect conductivity.

It is important to obtain a specific conductance measurement soon after taking a sample because temperature changes, precipitation reactions, and absorption of carbon dioxide from the air all affect specific conductance. Most conductivity meters in use today display specific conductance in units of mS/cm, which is the conductivity normalized to a temperature of 25°C. These are the required units to be recorded on the groundwater sample log field form or equivalent electronic form.

6.5.2.2 Principles of Equipment Operation

An aqueous system containing ions will conduct an electric current. In a direct-current field, the positive ions migrate toward the negative electrode, and the negatively charged ions migrate toward the positive electrode. Most inorganic acids, bases, and salts such as hydrochloric acid, sodium carbonate, and sodium chloride, respectively, are relatively good conductors. Conversely, organic compounds such as sucrose or benzene, which do not dissociate in aqueous solution, conduct a current very poorly if at all.

A conductance cell and a Wheatstone Bridge (for the measurement of potential difference) may be used for measurement of electrical resistance. The ratio of current applied to voltage across the cell may also

be used as a measure of conductance. The core element of the apparatus is the conductivity cell containing the solution of interest. Depending on the ionic strength of the aqueous solution to be tested, a potential difference is developed across the cell, which can be converted directly or indirectly (depending on instrument type) to a measurement of specific conductance.

6.5.2.3 Equipment

The following equipment is needed for taking specific conductance measurements:

- Stand-alone portable conductivity meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Calibration solution, as specified by the manufacturer.
- Manufacturer's operation manual.

A variety of conductivity meters are available that may also be used to monitor salinity and temperature. Probe types and cable lengths vary, so equipment must be obtained to meet the specific requirements of the sampling program.

6.5.2.4 Measurement Techniques for Specific Conductance

The steps involved in taking specific conductance measurements are as follows (calibration shall be conducted according to manufacturer's instructions):

1. Check batteries and calibrate instrument before going into the field.
2. Calibrate on a daily use basis (or as recommended by manufacturer), according to the manufacturer's instructions and record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form. Potassium chloride solutions with a specific conductance closest to the values expected in the field shall be used for calibration.
3. Rinse the cell with one or more portions of the sample to be tested or with deionized water and shake excess water from the cell.
4. Immerse the electrode in the sample and measure the conductivity.
5. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form.
6. Rinse the electrode with deionized water.

If the specific conductance measurements become erratic, recalibrate the instrument and see the manufacturer's instructions for troubleshooting assistance.

6.5.3 Measurement of Temperature

6.5.3.1 General

In combination with other parameters, temperature can be a useful indicator of the likelihood of biological action in a water sample. It can also be used to trace the flow direction of contaminated groundwater. Temperature measurements shall be taken in situ, or as quickly as possible in the field because collected water samples may rapidly equilibrate with the temperature of their surroundings.

6.5.3.2 Equipment

Temperature measurements may be taken with alcohol-toluene, mercury-filled, dial-type thermometers or combination meters equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22). In addition, various meters such as specific conductance or DO meters that have temperature measurement capabilities may also be used. Using such instrumentation along with suitable probes and cables, in-situ measurements of temperature at great depths can be performed.

6.5.3.3 Measurement Techniques for Water Temperature

If a thermometer is used to determine the temperature for a water sample, use the following procedure:

1. Immerse the thermometer in the sample until temperature equilibrium is obtained (1 to 3 minutes). To avoid the possibility of cross-contamination, the thermometer shall not be inserted into samples that will undergo subsequent chemical analysis.

2. Record values in a field logbook or on a sample log sheet or equivalent electronic form.

If a temperature meter or probe is used:

1. Calibrate the instrument according to manufacturer's recommendations prior to use.

2. Immerse the meter/probe in the sample until temperature equilibrium is obtained (1 to 3 minutes). To avoid the possibility of cross-contamination, the meter/probe shall not be inserted into samples that will undergo subsequent chemical analysis.

3. Record values in a field logbook or on a sample log sheet or equivalent electronic form.

6.5.4 Measurement of Dissolved Oxygen

6.5.4.1 General

DO levels in natural water and wastewater depend on the physical, chemical and biochemical activities in the water body. In addition, the growth of many aquatic organisms and the rate of corrosivity are dependent on DO concentrations. Thus, analysis for DO is a key test in water pollution and waste treatment process control. If at all possible, DO measurements shall be taken in situ because concentrations may show a large change in a short time if the sample is not adequately preserved.

The monitoring method discussed herein is limited to the use of DO meters. Chemical methods of analysis (i.e., Winkler methods) are available but require more equipment and greater sample manipulation. Furthermore, DO meters using a membrane electrode are suitable for highly polluted waters because the probe is completely submersible and is not susceptible to interference caused by color, turbidity, or colloidal material or suspended matter.

6.5.4.2 Principles of Equipment Operation

DO probes are normally electrochemical cells that have two solid metal electrodes of different nobility immersed in an electrolyte. The electrolyte is retained by an oxygen-permeable membrane. The metal of highest nobility (the cathode) is positioned at the membrane. When a suitable potential exists between the two metals, reduction of oxygen to hydroxide ion (OH^-) occurs at the cathode surface. An electrical

current is developed that is directly proportional to the rate of arrival of oxygen molecules at the cathode. This rate is proportional to the oxygen concentration in the water being measured.

Because the current produced in the probe is directly proportional to the rate of arrival of oxygen at the cathode, it is important that a fresh supply of sample always be in contact with the membrane. Otherwise, the oxygen in the aqueous layer along the membrane is quickly depleted and false low readings are obtained. It is therefore necessary to stir the sample (or the probe) constantly to maintain fresh solution near the membrane interface. Stirring, however, shall not be so vigorous that additional oxygen is introduced through the air-water interface at the sample surface. To avoid this possibility, some probes are equipped with stirrers to agitate the solution near the probe, leaving the surface of the solution undisturbed.

DO probes are relatively unaffected by interferences. Interferences that can occur are reactions with oxidizing gases such as chlorine or with gases such as hydrogen sulfide that are not easily depolarized from the indicating electrode. If a gaseous interference is suspected, it shall be noted in the field logbook and checked if possible. Temperature variations can also cause interference because probes exhibit temperature sensitivity. Automatic temperature compensation is normally provided by the manufacturer. This compensation can counteract some of the temperature effects but not all of them.

6.5.4.3 Equipment

The following equipment is needed to measure DO concentrations:

- A stand-alone portable DO meter or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Sufficient cable to allow the probe to contact the sample.
- Manufacturer's operation manual.

6.5.4.4 Measurement Techniques for Dissolved Oxygen Determination

DO probes differ as to instructions for use. Follow the manufacturer's instructions to obtain an accurate reading. The following general steps shall be used to measure DO concentrations:

1. Check the DO meter batteries before going to the field.
2. Condition the probe in a water sample for as long a period as practical before use in the field. Long periods of dry storage followed by short periods of use in the field may result in inaccurate readings.
3. Calibrate the instrument in the field according to manufacturer's recommendations or in a freshly air-saturated water sample of known temperature.
4. Record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.
5. Rinse the probe with deionized water.
6. Immerse the probe in the sample. Be sure to provide for sufficient flow past the membrane by stirring the sample. Probes without stirrers placed in wells may be moved up and down to achieve the required mixing.

7. Record the DO content and temperature of the sample in a field logbook or on a sample log sheet or equivalent electronic form.

8. Rinse the probe with deionized water.

9. Recalibrate the probe when the membrane is replaced, or as needed. Follow the manufacturer's instructions.

Note that in-situ placement of the probe is preferable because sample handling is not involved. This however may not always be practical.

Special care shall be taken during sample collection to avoid turbulence that can lead to increased oxygen solubilization and positive test interferences.

6.5.5 Measurement of Oxidation-Reduction Potential

6.5.5.1 General

ORP provides a measure of the tendency of organic or inorganic chemicals to exist in an oxidized state. The ORP parameter therefore provides evidence of the likelihood of anaerobic degradation of biodegradable organics or the ratio of activities of reduced to oxidized species in the sample.

6.5.5.2 Principles of Equipment Operation

When an inert metal electrode, such as platinum, is immersed in a solution, a potential is developed at that electrode depending on the ions present in the solution. If a reference electrode is placed in the same solution, an ORP electrode pair is established. This electrode pair allows the potential difference between the two electrodes to be measured and is dependent on the concentration of the ions in solution. By this measurement, the ability to oxidize or reduce species in solution may be determined. Supplemental measurements, such as DO, may be correlated with ORP to provide knowledge of the quality of the solution, water, or wastewater.

6.5.5.3 Equipment

The following equipment is needed for measuring the ORP of a solution:

- A combination meter with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Reference solution as specified by the manufacturer.
- Manufacturer's operation manual.

6.5.5.4 Measurement Techniques for Oxidation-Reduction Potential

The following procedure is used for measuring ORP:

1. Check the equipment using the manufacturer's recommended reference solution and check its batteries before going to the field.

2. Thoroughly rinse the electrode with deionized water.

3. If the probe does not respond properly to the recommended reference solution, verify the sensitivity of the electrodes by noting the change in millivolts when the pH of a test solution is altered. The ORP will increase when the pH of a test solution decreases, and the ORP will decrease when the test solution pH is increased. Place the sample in a clean container and agitate the sample. Insert the electrodes and note that the ORP drops sharply when the caustic is added (i.e., pH increases) thus indicating that the electrodes are sensitive and operating properly. If the ORP increases sharply when the caustic is added, the polarity is reversed and must be corrected in accordance with the manufacturer's instructions or the probe should be replaced.

4. Record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.

6.5.6 Measurement of Salinity

6.5.6.1 General

Salinity is a unitless property of industrial and natural waters. It is the measurement of dissolved salts in a given mass of solution. Most field meters determine salinity automatically from conductivity and temperature. The displayed value will be displayed in either parts per thousand (ppt) or percent (e.g., 35 ppt equals 3.5 percent).

6.5.6.2 Principles of Equipment Operation

Salinity is determined automatically from the meter's conductivity and temperature readings according to algorithms (such as are found in Standard Methods for the Examination of Water and Wastewater). Depending on the meter, the results are displayed in either ppt or percent. The salinity measurements are carried out in reference to the conductivity of standard seawater (corrected to salinity = 35 ppt).

6.5.6.3 Equipment

The following equipment is needed for salinity measurements:

- A multi-parameter water quality meter capable of measuring conductivity and temperature and converting them to salinity (e.g., Horiba U-22 or YSI 600 series).
- Calibration solution as specified by the manufacturer.
- Manufacturer's operation manual.

6.5.6.4 Measurement Techniques for Salinity

The steps involved in taking salinity measurements are as follows (standardization shall be conducted according to manufacturer's instructions):

1. Check the expiration date of the solutions used for field calibration and replace them if they are expired.

2. Check batteries and calibrate the meter before going into the field.

3. Calibrate on a daily use basis, according to the manufacturer's instructions and record all pertinent information on a water quality meter calibration log sheet or equivalent electronic form.

4. Rinse the cell with the sample to be tested. This is typically accomplished as the probe is placed in line during the collection of the purge water up to the time of sample acquisition.

5. Immerse the multi-probe in the sample and measure the salinity. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form.

6. Rinse the probes with deionized water.

6.5.7 Measurement of Turbidity

6.5.7.1 General

Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample. Turbidity in water is caused by suspended matter such as clay, silt, or other finely divided organic and inorganic matter and microscopic organisms including plankton.

It is important to obtain a turbidity reading immediately after taking a sample because irreversible changes in turbidity may occur if the sample is stored too long.

6.5.7.2 Principles of Equipment Operation

Turbidity is measured by the Nephelometric Method, which is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions. The higher the scattered light intensity, the higher the turbidity.

Formazin polymer is used as the reference turbidity standard suspension because of its ease of preparation combined with a higher reproducibility of its light-scattering properties than clay or turbid natural water. The turbidity of a specified concentration of formazin suspension is defined as 40 nephelometric units. This same suspension has an approximate turbidity of 40 Jackson units when measured on the candle turbidimeter. Therefore, nephelometric turbidity units (NTUs) based on the formazin preparation will approximate units derived from the candle turbidimeter but will not be identical to them.

6.5.7.3 Equipment

The following equipment is needed for turbidity measurements:

- A turbidity meter (e.g., LaMotte 2020) that calibrates easily using test cells with standards of 0.0, 1.0, and 10 NTUs, or a combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Calibration solution and sample tubes, as specified by the manufacturer.
- Manufacturer's operation manual.

6.5.7.4 Measurement Techniques for Turbidity

The steps involved in taking turbidity measurements utilizing an electrode (e) or light meter (l) are listed below (standardization shall be done according to manufacturer's instructions):

1. Check the expiration date of the solutions used for field calibration and replace them if they are expired.
2. Check batteries and calibrate the instrument before going into the field.
3. Calibrate on a daily basis according to the manufacturer's instructions, and record all pertinent information on a turbidity meter calibration log sheet (Attachment C) or equivalent electronic form.
4. When using the YSI and/or Horiba U-22, rinse the electrode with one or more portions of the sample to be tested or with deionized water.
5. When using the Lamotte 2020, fill the light meter's glass test cell with approximately 5 mL of sample, screw on the cap, wipe off glass to remove all residue that could intercept the instrument's light beam, place the test cell in the light meter, and close the lid.
6. Immerse the electrode in the sample and measure the turbidity.
7. The reading must be taken immediately because suspended solids will settle over time resulting in a lower, inaccurate turbidity reading.
8. Read and record the results in a field logbook or on a sample log sheet or equivalent electronic form. Include a physical description of the sample, including color, qualitative estimate of turbidity, etc.
9. Rinse the electrode or test cell with deionized water.

6.6 Sampling

6.6.1 Sampling Plan

The sampling approach consisting of the following shall be developed as part of the project planning documents approved prior to beginning work in the field:

- Background and objectives of sampling.
- Brief description of area and waste characterization.
- Identification of sampling locations, with map or sketch, and applicable well construction data (well size, depth, screened interval, reference elevation).
- Intended number, sequence, volumes, and types of samples. If the relative degree of contamination between wells is insignificant, a sampling sequence that facilitates sampling logistics may be followed. Where some wells are known or strongly suspected of being highly contaminated, these shall be sampled last to reduce the risk of cross-contamination between wells. In situations where the well is not well-characterized and the nature or extent of airborne contamination is unknown, it is recommended that head space analysis using a photoionization detector (PID) or flame ionization detector (FID) is performed to rate the wells, sampling from least contaminated to most contaminated.

Refer to the project-specific HASP for appropriate information and direction on air monitoring requirements.

- Sample preservation requirements.
- Work schedule.
- List of team members.
- List of observers and contacts.
- Other information, such as the necessity for a warrant or permission of entry, requirements for split samples, access problems, location of keys, etc.
- The FOL shall ensure that the sampling method(s) to be employed is accurately represented in the HASP, indicating the types of sampling to be employed and the hazards. If the methods are not accurately represented, the FOL should rectify this with the HASP author.
- The FOL shall ensure that sampling teams understand the sampling approach that they are to follow. Where sampling teams are made up of personnel from multiple locations, personal sampling experiences may vary. Therefore the FOL shall review project-specific requirements, SOPs, and protocol to be followed. The FOL will conduct periodic surveys to ensure that these methods are being completed per his/her direction.

6.6.2 Sampling Methods as Related to Low-Flow Sampling

The collection of a groundwater sample consists of the following steps:

1. Ensure the safety of the sample location. Take a few minutes to evaluate the area for physical hazards (trip hazards, uneven ground, overhanging branches, etc.) and natural hazards (snakes, bees, spiders, etc.) that may exist in the area or that may have constructed nests in the well head. Snakes often like to sun themselves on concrete well pads. Follow provisions in the project-specific HASP and/or HSGM for addressing natural hazards.
2. As indicated earlier, some monitoring wells have the potential to contain pressurized headspace (e.g., through the generation of gases form contaminated groundwater, due to biological processes, degradation of contaminants, or simply based on location such as near a landfill or in areas that intersect lithological abnormalities) or through intentional artificial means such as those associated with air sparging systems. Injection or extraction wells may be artificially pressurized and may remain so for several days after the system has been turned off. This presents a hazard to people opening these wells. The Field Sampling Technician shall employ the following practices to minimize these hazards:

    - Wear safety glasses to protect the eyes. If site-specific observations and conditions indicate that the wells may be pressurized, wear a full-face shield over the safety impact eye protection.
    - DO NOT place your face or any other part of your body over the well when opening because this may place you in a strike zone.
    - Open the well cover at arms length, then step away and allow the well to off gas and stabilize.

    Follow directions provided in the project-specific HASP, Work Plan and/or Sampling Plan pertaining to the use of volatile chemical detection equipment (PID or FID) within the breathing zone of the

sampler during sampling to determine the need to retreat from the work area and/or for the use of respiratory protection (as specified in the HASP).

3. When proper respiratory protection has been donned, sound the well for total depth and water level (using clean equipment) and record these data on a groundwater sampling log sheet or equivalent electronic form; then calculate the fluid volume in the well pipe (as previously described in this SOP). It is imperative that downhole equipment be adequately decontaminated between wells to prevent cross-contamination. Just as sampling occurs from the least contaminated to the most contaminated, it is also recommended that groundwater level measurements be taken in this manner.

4. Calculate volume of well water to be removed as described in Section 6.3.

5. Select the appropriate purging equipment (see Attachment A to this SOP) or as designated within your Work Plan/Sampling Plan. If an electric submersible pump with packer is chosen, go to Step 10.

6. Lower the purging equipment or intake into the well to a short distance below the water level or mid-screen as indicated in project-specific documentation and begin water removal. Remember that some contaminants are "bottom dwellers," and in these cases, project-specific direction may specify placing the intake just above (1 to 2 feet) the well bottom. Secure the pump intake at the well and secure the effluent at the collection container and begin pumping. The pumping rate will be determined based on the decrease in the water level (see Section 6.7) or as directed in your project-specific documents or this SOP. Purge water is generally collected in a 5-gallon bucket or similar open- or closed-top container. To minimize the potential for spills and back injuries, do not fill 5-gallon buckets beyond approximately 80 percent of their capacity. Dispose of purge water was as indicated in the planning document(s). Where necessary, slow the pumping rate or lower the pump intake as required to maintain submergence.

7. Estimate the approximate rate of discharge frequently and record it on the Low Flow Purge Data Sheet (see Attachment D). Estimate flow rate by noting the amount of discharge in a bucket or graduated cylinder per unit time using a watch with a second hand or a stopwatch.

8. Observe the peristaltic pump tubing intake for degassing "bubbles." If bubbles are abundant and the intake is fully submerged, this pump is not suitable for collecting samples for volatile organics.

9. Purge a minimum of three to five casing volumes before sampling (or as directed by the site-specific SAP). In low-permeability strata (i.e., if the well is pumped to dryness), one volume will suffice. Allow the well to recover to 75 percent of initial water level before sampling. Do not overfill purge containers because this increases the potential for spills and lifting injuries.

10. If sampling using a submersible pump, lower the pump intake to mid-screen (or the middle of the open section in uncased wells) and collect the sample. If sampling with a bailer, lower the bailer to just below the water surface.

11. For pump and packer assemblies only: Lower the assembly into the well so that the packer is positioned just above the screen or open section. Inflate the packer. Purge a volume equal to at least twice the screened interval (or unscreened open section volume below the packer) before sampling. Packers shall always be tested in a casing section above ground to determine proper inflation pressures for good sealing.

12. If the recovery time of the well is very slow (e.g., 24 hours or greater), sample collection can be delayed until the following day. If the well has been purged early in the morning, sufficient water may be standing in the well by the day's end to permit sample collection. If the well is incapable of producing a sufficient volume of sample at any time, take the largest quantity available and record

this occurrence in the site logbook or equivalent electronic form. When this occurs, contact the analytical laboratory to alert them that a reduced sample volume(s) will be submitted for analysis.

13. Fill sample containers and preserve and label them as described in SOP SA-6.1. Many sample bottles will contain preservative when they are shipped to the field. In those cases, do not add preservative.

14. Replace the well cap and lock it as appropriate. Make sure the well is readily identifiable as the source of the sample.

15. Process sample containers as described in SOP SA-6.1.

16. Decontaminate equipment as described in SOP SA-7.1.

6.7 Low-Flow Purging and Sampling

6.7.1 Scope and Application

Low-flow purging and sampling techniques may be required for groundwater sampling activities. The purpose of low-flow purging and sampling is to collect groundwater samples that contain "representative" amounts of mobile organic and inorganic constituents in the vicinity of the selected open well interval, at or near natural flow conditions. This minimum-stress procedure emphasizes negligible water level drawdown and low pumping rates to collect samples with minimal alterations in water chemistry. This procedure is designed primarily to be used in wells with a casing diameter of 1 inch or more and a saturated screen length, or open interval, of 10 feet or less. Samples obtained are suitable for analyses of common types of groundwater contaminants (volatile and semivolatile organic compounds, pesticides, polychlorinated biphenyls [PCBs], metals and other inorganic ions [cyanide, chloride, sulfate, etc.]). This low-flow procedure is not designed for collection of non-aqueous phase liquid samples from wells containing light or dense non-aqueous phase liquids (LNAPLs or DNAPLs).

This procedure is flexible for various well construction types and groundwater yields. The goal of the procedure is to obtain a turbidity level of less than 10 NTUs and to achieve a water level drawdown of less than 0.3 foot during purging and sampling. If these goals cannot be achieved, sample collection can take place provided that the remaining criteria in this procedure are met.

6.7.2 Equipment

The following equipment is required (as applicable) for low-flow purging and sampling:

- Adjustable rate submersible pump (e.g., centrifugal or bladder pump constructed of stainless steel or Teflon).

- Disposable clear plastic bottom-filling bailers to be used to check for and obtain samples of LNAPLs or DNAPLs.

- Tubing – Teflon, Teflon-lined polyethylene, polyethylene, polyvinyl chloride (PVC), Tygon, or stainless steel tubing can be used to collect samples for analysis, depending on the analyses to be performed and regulatory requirements.

- Water level measuring device with 0.01-foot accuracy (electronic devices are preferred for tracking water level drawdown during all pumping operations).

- Interface probe.
- Flow measurement supplies.
- Power source (generator, nitrogen tank, etc.). If a gasoline generator is used, it must be located downwind and at a safe distance from the well so that the exhaust fumes do not contaminate the samples.
- Indicator parameter monitoring instruments – pH, turbidity, specific conductance, and temperature. Use of a flow-through cell is recommended. Optional indicators - ORP, salinity, and DO. A flow-through cell (also referred to as an in-line sample chamber) is required.
- Standards to perform field calibration of instruments.
- Decontamination supplies.
- Logbook(s) and other forms (see Attachments B through D) or equivalent electronic form(s).
- Sample bottles.
- Sample preservation supplies (as required by the analytical methods).
- Sample tags and/or labels.
- Well construction data, location map, field data from last sampling event (if available).
- Field Sampling Plan.
- PID or FID instrument for measuring volatile organic compounds (VOCs) per the HASP.

6.7.3 Purging and Sampling Procedure

1. Open the monitoring well as stated earlier and step away. Prepare sampling equipment while allowing 3 to 5 minutes to allow the water level to reach equilibrium. In situations where VOCs are the primary contaminants of concern, air monitoring of the samplers' breathing zone areas may be required by the HASP (typically with a PID or FID).

2 Measure the water level immediately prior to placing the pump in the well and record the water level on the Low-Flow Purge Data Form or equivalent electronic form immediately prior to placing the pump or tubing into the well.

3. Lower the measuring device further into the well to collect the total depth measurement. Again wait 3 to 5 minutes to allow the well to equilibrate to the initial water level prior to placing the pump or pump intake in the well.

4. Record the total well depth on the Low-Flow Purge Data Form or equivalent electronic form immediately prior to placing the pump or tubing into the well

5. Lower the pump or tubing slowly into the well so that the pump intake is located at the center of the saturated screen length of the well. If possible, keep the pump intake at least 2 feet above the bottom of the well to minimize mobilization of sediment that may be present in the bottom of the well. Collection of turbidity-free water samples may be difficult if there is 3 feet or less of standing water in the well.

6. Start with the initial pump rate set at approximately 0.1 liter per minute. Use a graduated cylinder and stopwatch to measure the pumping rate. Adjust the pumping rates as necessary to prevent drawdown from exceeding 0.3 foot during purging. If no drawdown is noted, the pump rate may be increased (to a maximum of 0.4 liter per minute) to expedite the purging and sampling event. The pump rate will be reduced if turbidity is greater than 10 NTUs after all other field parameters have stabilized. If groundwater is drawn down below the top of the well screen, purging shall cease or the well shall be pumped to dryness and then allowed to recover before purging continues. Well recovery to 75 percent is necessary prior to sampling. Slow-recovering wells should be identified and purged at the beginning of the workday to maximize field work efficiency. If possible, samples should be collected from these wells within the same workday and no later than 24 hours after the end of purging.

7. Measure the water level in the well every 5 to 10 minutes using the water level meter. Record the well water level on the Low Flow Purge Data Form (Attachment D) or equivalent electronic form.
8. Record on the Low Flow Purge Data Form every 5 to 10 minutes the water quality parameters (pH, specific conductance, temperature, turbidity, ORP, DO, and salinity or as specified by the approved site-specific planning document) measured by the water quality meter and turbidity meter. If the cell needs to be cleaned during purging operations, continue pumping (allow the pump to discharge into a container) and disconnect the cell. Rinse the cell with distilled/deionized water. After cleaning is completed, reconnect the flow-through cell and continue purging. Document the cell cleaning on the Low-Flow Purge Data Form or equivalent electronic form.

9. Estimate the flow rate by noting the amount of discharge in a graduated cylinder per unit time using a watch with a second hand. Remeasure the flow rate any time the pump rate is adjusted and periodically during purging. This will determine if a reduction in rate has occurred due to possible battery depletion.

10. During purging, check for the presence of bubbles in the flow-through cell. The presence of bubbles is an indication that connections are not tight. If bubbles are observed, check for loose connections and tighten, repair, or replace them as necessary to achieve a tight connection.

11. Wait until stabilization is achieved, or a minimum of two saturated screen volumes have been removed and three consecutive readings, taken at 5 to 10 minute intervals, are within the following limits, then begin sampling:

    - pH ±0.2 standard units
    - Specific conductance ±10%
    - Temperature ±10%
    - Turbidity less than 10 NTUs
    - DO ±10%

12. If the above conditions have not been met after the well has been purged for 4 hours, purging will be considered complete and sampling can begin. Record the final well stabilization parameters from the Low-Flow Purge Data Form onto the Groundwater Sample Log Form or equivalent electronic form.

NOTE: VOC samples are preferably collected first, directly into pre-preserved sample containers. Fill all sample containers by allowing the pump discharge to flow gently down the inside of the container with minimal turbulence.

13. If the water column in the pump tubing collapses (water does not completely fill the tubing) before exiting the tubing, use one of the following procedures to collect VOC samples:

- Collect samples for non-VOC analyses first, then increase the flow rate incrementally until the water column completely fills the tubing, collect the sample for VOCs, and record the new flow rate.

- Reduce the diameter of the existing tubing until the water column fills the tubing either by adding a connector (Teflon or stainless steel) or clamp, which should reduce the flow rate by constricting the end of the tubing. Proceed with sample collection.

- Insert a narrow-diameter Teflon tube into the pump's tubing so that the end of the tubing is in the water column and the other end of the tubing protrudes beyond the pump's tubing, then collect the sample from the narrow diameter tubing.

- Prepare samples for shipping as per SOP SA-6.1.

7.0 REFERENCES

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ATTACHMENT A

PURGING EQUIPMENT SELECTION

Diameter Casing		Bailer	Peristaltic Pump	Vacuum Pump	Air-lift	Diaphragm "Trash" Pump	Submersible Diaphragm Pump	Submersible Electric Pump	Submersible Electric Pump w/Packer
1.25-Inch	Water level <25 feet	X	X	X	X	X			
	Water Level >25 feet	X			X				
2-Inch	Water level <25 feet	X	X	X	X	X	X		
	Water Level >25 feet	X			X		X		
4-Inch	Water level <25 feet	X	X	X	X	X	X	X	X
	Water Level >25 feet	X			X		X	X	X
6-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X
8-Inch	Water level <25 feet				X	X		X	X
	Water Level >25 feet				X			X	X

ATTACHMENT A

PURGING EQUIPMENT SELECTION

PAGE 2

Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/Length (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
BarCad Systems, Inc.	BarCad Sampler	Dedicated; gas drive (positive displacement)	1.5/16	PE, brass, nylon, aluminum oxide	0-150 with std. tubing	1 liter for each 10-15 feet of submergence	$220-350	Requires compressed gas; custom sizes and materials available; acts as piezometer.
Cole-Parmer Inst. Co.	Master Flex 7570 Portable Sampling Pump	Portable; peristaltic (suction)	<1.0/NA	(not submersible) Tygon®, silicone Viton®	0-30	670 mL/min with 7015-20 pump head	$500-600	AC/DC; variable speed control available; other models may have different flow rates.
ECO Pump Corp.	SAMPLifier	Portable; venturi	<1.5 or <2.0/NA	PP, PE, PVC, SS, Teflon®, Tefzel®	0-100	0-500 mL/min depending on lift	$400-700	AC, DC, or gasoline-driven motors available; must be primed.
Geltek Corp.	Bailer 219-4	Portable; grab (positive displacement)	1.66/38	Teflon®	No limit	1,075 mL	$120-135	Other sizes available.
GeoEngineering, Inc.	GEO-MONITOR	Dedicated; gas drive (positive displacement)	1.5/16	PE, PP, PVC, Viton®	Probably 0-150	Approximately 1 liter for each 10 feet of submergence	$185	Acts as piezometer; requires compressed gas.
Industrial and Environmental Analysts, Inc. (IEA)	Aquarius	Portable; bladder (positive displacement)	1.75/43	SS, Teflon®, Viton®	0-250	0-2,800 mL/min	$1,500-3,000	Requires compressed gas; other models available; AC, DC, manual operation possible.
IEA	Syringe Sampler	Portable; grab (positive displacement)	1.75/43	SS, Teflon®	No limit	850 mL sample volume	$1,100	Requires vacuum and/or pressure from hand pump.
Instrument Specialties Co. (ISCO)	Model 2600 Well Sampler	Portable; bladder (positive displacement)	1.75/50	PC, silicone, Teflon®, PP, PE, Detrin®, acetal	0-150	0-7,500 mL/min	$990	Requires compressed gas (40 psi minimum).
Keck Geophysical Instruments, Inc.	SP-81 Submersible Sampling Pump	Portable; helical rotor (positive displacement)	1.75/25	SS, Teflon®, PP, EPDM, Viton®	0-160	0-4,500 mL/min	$3,500	DC operated.
Leonard Mold and Die Works, Inc.	GeoFilter Small Diameter Well Pump (#0500)	Portable; bladder (positive displacement)	1.75/38	SS, Teflon®, PC, Neoprene®	0-400	0-3,500 mL/min	$1,400-1,500	Requires compressed gas (55 psi minimum); pneumatic or AC/DC control module.
Oil Recovery Systems, Inc.	Surface Sampler	Portable; grab (positive displacement)	1.75/12	acrylic, Detrin®	No limit	Approximately 250 mL	$125-160	Other materials and models available; for measuring thickness of "floating" contaminants.
Q.E.D. Environmental Systems, Inc.	Well Wizard® Monitoring System (P-100)	Dedicated; bladder (positive displacement)	1.66/36	PVC	0-230	0-2,000 mL/min	$300-400	Requires compressed gas; piezometric level indicator; other materials available.

ATTACHMENT A

PURGING EQUIPMENT SELECTION

PAGE 3

Manufacturer	Model Name/Number	Principle of Operation	Maximum Outside Diameter/Length (Inches)	Construction Materials (w/Lines and Tubing)	Lift Range (ft)	Delivery Rates or Volumes	1982 Price (Dollars)	Comments
Randolph Austin Co.	Model 500 Vari-Flow Pump	Portable; peristaltic (suction)	<0.5/NA	(Not submersible) Rubber, Tygon®, or Neoprene®	0-30	See comments	$1,200-1,300	Flow rate dependent on motor and tubing selected; AC operated; other models available.
Robert Bennett Co.	Model 180	Portable; piston (positive displacement)	1.8/22	SS, Teflon®, Delrin® PP, Viton®, acrylic, PE	0-500	0-1,800 mL/min	$2,600-2,700	Requires compressed gas; water level indicator and flow meter; custom models available.
Slope Indicator Co. (SINCO)	Model 514124 Pneumatic Water Sampler	Portable; gas drive (positive displacement)	1.9/18	PVC, nylon	0-1,100	250 mL/flushing cycle	$250-350	Requires compressed gas; SS available; piezometer model available; dedicated model available.
Solinst Canada Ltd.	5W Water Sampler	Portable; grab (positive displacement)	1.9/27	PVC, brass, nylon, Neoprene®	0-330	500 mL	$1,300-1,800	Requires compressed gas; custom models available.
TIMCO Mfg. Co., Inc.	Std. Bailer	Portable; grab (positive displacement)	1.66/Custom	PVC, PP	No limit	250 mL/ft of bailer	$20-60	Other sizes, materials, models available; optional bottom-emptying device available; no solvents used.
TIMCO	Air or Gas Lift Sampler	Portable; gas drive (positive displacement)	1.66/30	PVC, Tygon®, Teflon®	0-150	350 mL/flushing cycle	$100-200	Requires compressed gas; other sizes, materials, models available; no solvents used.
Tole Devices Co.	Sampling Pump	Portable; bladder (positive displacement)	1.38/48	SS, silicone, Delrin®, Tygon®	0-125	0-4,000 mL/min	$800-1,000	Compressed gas required; DC control module; custom built.

Construction Material Abbreviations:

PE	Polyethylene
PP	Polypropylene
PVC	Polyvinyl chloride
SS	Stainless steel
PC	Polycarbonate
EPDM	Ethylene-propylene diene (synthetic rubber)

Other Abbreviations:

NA	Not applicable
AC	Alternating current
DC	Direct current

NOTE: Other manufacturers market pumping devices which could be used for groundwater sampling, though not expressly designed for this purpose. The list is not meant to be all-inclusive and listing does not constitute endorsement for use. Information in the table is from sales literature and/or personal communication. No skimmer, scavenger-type, or high-capacity pumps are included.

Source: Barcelona et al., 1983.

ATTACHMENT B
GROUNDWATER SAMPLE LOG SHEET

Tetra Tech

GROUNDWATER SAMPLE LOG SHEET

Page___ of___

Project Site Name: _____
Project No.: _____

[ ] Domestic Well Data
[x] Monitoring Well Data
[ ] Other Well Type: _____
[ ] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____
Type of Sample:
[X] Low Concentration
[ ] High Concentration

SAMPLING DATA:

Date:	Color	pH	S.C.	Temp.	Turbidity	DO	ORP	Other
Time:	Visual	Standard	mS/cm	Degrees C	NTU	mg/l	mV	NA
Method:								

PURGE DATA:

Date:	Volume	pH	S.C.	Temp. (C)	Turbidity	DO	ORP	Other
Method:								
Monitor Reading (ppm):			SEE LOW FLOW PURGE DATA SHEET					
Well Casing Diameter & Material								
Type:								
Total Well Depth (TD):								
Static Water Level (WL):								
One Casing Volume(gal/L):								
Start Purge (hrs):								
End Purge (hrs):								
Total Purge Time (min):								
Total Vol. Purged (gal/L):								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

Circle if Applicable:		Signature(s):
MS/MSD	Duplicate ID No.:	

ATTACHMENT C
EQUIPMENT CALIBRATION LOG

Tetra Tech

EQUIPMENT CALIBRATION LOG

PROJECT NAME : _____ INSTRUMENT NAME/MODEL: _____

SITE NAME: _____ MANUFACTURER: _____

PROJECT No.: _____ SERIAL NUMBER: _____

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

ATTACHMENT D
LOW FLOW PURGE DATA SHEET

LOW FLOW PURGE DATA SHEET

PROJECT SITE NAME: ____________ WELL ID: ____________

PROJECT NUMBER: ____________ DATE: ____________

Time (Hrs.)	Water Level (Ft. below TOC)	Flow (mL/Min.)	pH (S.U.)	S. Cond. (mS/cm)	Turb. (NTU)	DO (mg/L)	Temp. (Celcius)	ORP mV	Salinity % or ppt	Comments

SIGNATURE(S): ____________ PAGE___OF___

TETRA TECH	STANDARD OPERATING PROCEDURES	Number GH-2.8	Page 1 of 12
		Effective Date 01/2012	Revision 4
		Applicability Tetra Tech, Inc.	
		Prepared Earth Sciences Department	
Subject GROUNDWATER MONITORING WELL INSTALLATION		Approved J. Zimmerly	

TABLE OF CONTENTS

1.0 PURPOSE

This procedure provides general guidance and information pertaining to proper monitoring well design, installation, and development.

2.0 SCOPE

This procedure is applicable to the construction of monitoring wells. The methods described herein may be modified by project-specific requirements for monitoring well construction. In addition, many regulatory agencies have specific regulations pertaining to monitoring well construction and permitting. These requirements must be determined during the project planning phases of the investigation, and any required permits must be obtained before field work begins. Innovative monitoring well installation techniques, which typically are not used, will be discussed only generally in this procedure.

3.0 GLOSSARY

Monitoring Well - A well which is screened, cased, and sealed which is capable of providing a groundwater level and groundwater sample representative of the zone being monitored. Some monitoring wells may be constructed as open boreholes.

Piezometer - A pipe or tube inserted into the water bearing zone, typically open to water flow at the bottom and to the atmosphere at the top, and used to measure water level elevations. Piezometers may range in size from 1/2-inch-diameter plastic tubes to well points or monitoring wells.

Potentiometric Surface - The surface representative of the level to which water will rise in a well cased to the screened aquifer.

Well Point (Drive Point) - A screened or perforated tube (Typically 1-1/4 or 2 inches in diameter) with a solid, conical, hardened point at one end, which is attached to a riser pipe and driven into the ground with a sledge hammer, drop weight, or mechanical vibrator. Well points may be used for groundwater injection and recovery, as piezometers (i.e., to measure water levels) or to provide groundwater samples for water quality data.

4.0 RESPONSIBILITIES

Driller - The driller provides adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of performing all phases of proper monitoring well installation and construction. The driller may also be responsible for obtaining, in advance, any required permits for monitoring well installation and construction.

Field Geologist - The field geologist supervises and documents well installation and construction performed by the driller, and insures that well construction is adequate to provide representative groundwater data from the monitored interval. Geotechnical engineers, field technicians, or other suitable trained personnel may also serve in this capacity.

5.0 PROCEDURES

5.1 Equipment/Items Needed

Below is a list of items that may be needed when installing a monitoring well or piezometer:

- Health and safety equipment (hard hats, safety glasses, etc.) as required by the Site Safety Officer.
- Well drilling and installation equipment with associated materials (typically supplied by the driller).
- Hydrogeologic equipment (weighted engineer's tape, water level indicator, retractable engineers rule, electronic calculator, clipboard, mirror and flashlight - for observing downhole activities, paint and ink marker for marking monitoring wells, sample jars, well installation forms, and a field notebook).
- Drive point installation tools (sledge hammer, drop hammer, or mechanical vibrator; tripod, pipe wrenches, drive points, riser pipe, and end caps).

5.2 Well Design

The objectives and intended use for each monitoring well must be clearly defined before the monitoring system is designed. Within the monitoring system, different monitoring wells may serve different purposes and, therefore, require different types of construction. During all phases of the well design, attention must be given to clearly documenting the basis for design decisions, the details of well construction, and the materials used. The objectives for installing the monitoring wells may include:

- Determining groundwater flow directions and velocities.
- Sampling or monitoring for trace contaminants.
- Determining aquifer characteristics (e.g., hydraulic conductivity).

Siting of monitoring wells shall be performed after a preliminary estimation of the groundwater flow direction. In most cases, groundwater flow directions and potential well locations can be determined by an experienced hydrogeologist through the review of geologic data and the site terrain. In addition, data from production wells or other monitoring wells in the area may be used to determine the groundwater flow direction. If these methods cannot be used, piezometers, which are relatively inexpensive to install, may have to be installed in a preliminary investigative phase to determine groundwater flow direction.

5.2.1 Well Depth, Diameter, and Monitored Interval

The well depth, diameter, and monitored interval must be tailored to the specific monitoring needs of each investigation. Specification of these items generally depends on the purpose of the monitoring system and the characteristics of the hydrogeologic system being monitored. Wells of different depth, diameter, and monitored interval can be employed in the same groundwater monitoring system. For instance, varying the monitored interval in several wells, at the same location (cluster wells) can help to determine the vertical gradient and the depths at which contaminants are present. Conversely, a fully penetrating well is usually not used to quantify or vertically locate a contaminant plume, since groundwater samples collected in wells that are screened over the full thickness of the water-bearing zone will be representative of average conditions across the entire monitored interval. However, fully penetrating wells can be used to establish the existence of contamination in the water-bearing zone. The well diameter desired depends upon the hydraulic characteristics of the water-bearing zone, sampling requirements, drilling method and cost.

The decision concerning the monitored interval and well depth is based on the following (and possibly other) information:

- The vertical location of the contaminant source in relation to the water-bearing zone.
- The depth, thickness and uniformity of the water-bearing zone.
- The anticipated depth, thickness, and characteristics (e.g., density relative to water) of the contaminant plume.
- Fluctuation in groundwater levels (due to pumping, tidal influences, or natural recharge/discharge events).
- The presence and location of contaminants encountered during drilling.
- Whether the purpose of the installation is for determining existence or non-existence of contamination or if a particular stratigraphic zone is being investigated.
- The analysis of borehole geophysical logs.

In most situations where groundwater flow lines are horizontal, depending on the purpose of the well and the site conditions, monitored intervals are 20 feet or less. Shorter screen lengths (5 feet or less) are usually required where flow lines are not horizontal, (i.e., if the wells are to be used for accurate measurement of the potentiometric head at a specific point).

Many factors influence the diameter of a monitoring well. The diameter of the monitoring well depends on the application. In determining well diameter, the following needs must be considered:

- Adequate water volume for sampling.
- Drilling methodology.
- Type of sampling device to be used.
- Costs.

Standard monitoring well diameters are 2, 4, 6, or 8 inches. Drive points are typically 1-1/4 or 2 inches in diameter. For monitoring programs which require screened monitoring wells, either a 2-inch or 4-inch-diameter well is preferred. Typically, well diameters greater than 4 inches are used in monitoring programs in which open-hole bedrock monitoring wells are used. With smaller diameter wells, the volume of stagnant water in the well is minimized, and well construction costs are reduced; however, the sampling devices that can be used are limited.

In specifying well diameter, sampling requirements must be considered (up to a total of 4 gallons of water may be required for a single sample to account for full organic and inorganic analyses, and split samples), particularly if the monitored formation is known to be a low-yielding formation. The unit volume of water contained within a monitoring well is dependent on the well diameter as follows:

Casing Inside Diameter (Inch)	Standing Water Length to Obtain 1 Gallon Water (Feet)
2	6.13
4	1.53
6	0.68

If a well recharges quickly after purging, then well diameter may not be an important factor regarding sample volume requirements.

Pumping tests for determining aquifer characteristics may require larger diameter wells (for installation of high capacity pumps); however, in small-diameter wells in-situ permeability tests can be performed during drilling or after well installation is completed.

5.2.2 Riser Pipe and Screen Materials

Well materials are specified by diameter, type of material, and thickness of pipe. Well screens require an additional specification of slot size. Thickness of pipe is referred to as "Schedule" for polyvinyl chloride (PVC) casing and is usually Schedule 40 (thinner wall) or 80 (thicker wall). Steel pipe thickness is often referred to as "Strength". Standard Strength is usually adequate for monitoring well purposes. With larger diameter pipe, the wall thickness must be greater to maintain adequate strength. The required thickness is also dependent on the method of installation; risers for drive points require greater strength than wells installed inside drilled borings.

The selection of well screen and riser materials depends on the method of drilling, the type of subsurface materials the well penetrates, the type of contamination expected, and natural water quality and depth. Cost and the level of accuracy required are also important. The materials generally available are Teflon, stainless steel, PVC galvanized steel, and carbon steel. Each has advantages and limitations (see Attachment A of this guideline for an extensive presentation on this topic). The two most commonly used materials are PVC and stainless steel. Properties of these two materials are compared in Attachment B. Stainless steel is a good choice where trace metals or organic sampling is required; however, costs are high. Teflon materials are extremely expensive, but are relatively inert and provide the least opportunity for water contamination due to well materials. PVC has many advantages, including low cost, excellent availability, light weight, ease of manipulation, and widespread acceptance. The crushing strength of PVC may limit the depth of installation, but the use of Schedule 80 materials may overcome some of the problems associated with depth. However, the smaller inside diameter of Schedule 80 pipe may be an important factor when considering the size of bailers or pumps required for sampling or testing. Due to this problem, the minimum well pipe size recommended for Schedule 80 wells is 4-inch I.D.

Screens and risers may have to be decontaminated before use because oil-based preservatives and oil used during thread cutting and screen manufacturing may contaminate samples. Metal pipe may corrode and release metal ions or chemically react with organic constituents, but this is considered a minor issue. Galvanized steel is not recommended where samples may be collected for metals analyses, as zinc and cadmium levels in groundwater samples may become elevated from leaching of the zinc coating.

Threaded, flush-joint casing is most often preferred for monitoring well applications. PVC, Teflon, and steel can all be obtained with threaded joints. Welded-joint steel casing is also acceptable. Glued PVC may release organic contaminants into the well, and therefore, should not be used if the well is to be sampled for organic constituents.

When the water-bearing zone is in consolidated bedrock, such as limestone or fractured granite, a well screen is often not necessary (the well is simply an open hole in bedrock). Unconsolidated materials, such as sands, clay, and silts require a screen. A screen slot size of 0.010 or 0.020 inch is generally used when a screen is necessary, and the annular borehole space around the screened interval is artificially packed with an appropriately sized sand, selected based on formation grain size. The slot size controls the quantity of water entering the well and prevents entry of natural materials or sand pack. The screen shall pass no more than 10 percent of the pack material, or in-situ aquifer material. The site geologist shall specify the combination of screen slot size and sand pack which will be compatible with the water-bearing zone, to maximize groundwater inflow and minimize head losses and movement of fines into the wells. For example, as a standard procedure, a Morie No. 1 or No. 10 to No. 20 U.S. Standard Sieve size filter pack is typically appropriate for a 0.020-inch slot screen; however, a No. 20 to No. 40 U.S. Standard Sieve size filter pack is typically appropriate for a 0.010-inch slot screen.

5.2.3 Annular Materials

Materials placed in the annular space between the borehole and riser pipe and screen include a sand pack when necessary, a bentonite seal, and cement-bentonite grout. The sand pack is usually a medium-to coarse-grained poorly graded, silica sand and should relate to the grain size of the aquifer sediments. The quantity of sand placed in the annular space is dependent upon the length of the screened interval, but should always extend at least 1 foot above the top of the screen. At least 1 to 3 feet of bentonite pellets or equivalent shall be placed above the sand pack. Cement-bentonite grout (or equivalent) is then placed to extent from the top of the bentonite pellets to the ground surface.

On occasion, and with the concurrence of the involved regulatory agencies, monitoring wells may be packed naturally (i.e., no artificial sand pack installed). In this case, the natural formation material is allowed to collapse around the well screen after the well is installed. This method has been used where the formation material itself is a relatively uniform grain size, or when artificial sand packing is not possible due to borehole collapse.

Bentonite expands by absorbing water and provides a seal between the screened interval and the overlying portion of the annular space and formation. Cement-bentonite grout is placed on top of the bentonite pellets, extending to the surface. The grout effectively seals the remaining borehole annulus and eliminates the possibility for surface infiltration reaching the screened interval. Grouting also replaces material removed during drilling and prevents hole collapse and subsidence around the well. A tremie pipe should be used to introduce grout from the bottom upward, to prevent bridging, and to provide a better seal. In shallow boreholes that don't collapse, it may be more practical to pour the grout from the surface without a tremie pipe.

Grout is a general term which has several different connotations. For all practical purposes within the monitoring well installation industry, grout refers to the solidified material which is installed and occupies the annular space above the bentonite pellet seal. Grout, most of the time, is made up of one or two assemblages of material, (e.g., cement and/or bentonite). A cement-bentonite grout, which is the most common type of grout used in monitoring well completions, normally is a mixture of cement, bentonite, and water at a ratio of one 90-pound bag of Portland Type I cement, plus 3 to 5 pounds of granular or flake-type bentonite, and 6-7 gallons of water. A neat cement consists of one ninety-pound bag of Portland Type I cement and 6-7 gallons of water. A bentonite slurry (bentonite and water mixed to a thick but pumpable mixture) is sometimes used instead of grout for deep well installations where placement of bentonite pellets is difficult. Bentonite chips are also occasionally used for annular backfill in place of grout.

In certain cases, the borehole may be drilled to a depth greater than the anticipated well installation depth. For these cases, the well shall be backfilled to the desired depth with bentonite pellets/chips or sand. A short (1- to 2-foot) section of capped riser pipe sump is sometimes installed immediately below the screen, as a silt reservoir, when significant post-development silting is anticipated. This will ensure that the entire screen surface remains unobstructed.

5.2.4 Protective Casing

When the well is completed and grouted to the surface, a protective steel casing is typically placed over the top of the well. This casing generally has a hinged cap and can be locked to prevent vandalism. The protective casing has a larger diameter than the well and is set into the wet cement grout over the well upon completion. In addition, one hole is drilled just above the cement collar through the protective casing which acts as a weep hole for the flow of water which may enter the annulus during well development, purging, or sampling.

A protective casing which is level with the ground surface (flush-mounted) is used in roadway or parking lot applications where the top of a monitoring well must be below the pavement. The top of the riser pipe is placed 4 to 5 inches below the pavement, and a locking protective casing is cemented in place to 3 inches below the pavement. A large diameter, manhole-type protective collar is set into the wet cement around the well with the top set level with or slightly above the pavement. An appropriately-sized id is placed over the protective sleeve. The cement should be slightly mounded to direct pooled water away from the well head.

5.3 Monitoring Well Installation

Pertinent data regarding monitoring well installation shall be recorded on log sheets as depicted and discussed in SOP SA-6.3. Attachments to this referenced SOP illustrate terms and physical construction of various types of monitoring wells.

5.3.1 Monitoring Wells in Unconsolidated Sediments

After the borehole is drilled to the desired depth, well installation can begin. The procedure for well installation will partially be dictated by the stability of the formation in which the well is being placed. If the borehole collapses immediately after the drilling tools are withdrawn, then a temporary casing must be installed and well installation will proceed through the center of the temporary casing, and continue as the temporary casing is withdrawn from the borehole. In the case of hollow-stem auger drilling, the augers will act to stabilize the borehole during well installation.

Before the screen and riser pipe are lowered into the borehole, all pipe and screen sections should be measured with an engineer's rule to ensure proper placement. When measuring sections, the threads on one end of the pipe or screen must be excluded while measuring, since the pipe and screen sections are screwed flush together.

After the screen and riser pipe are lowered through the temporary casing, the sand pack can be installed. A weighted tape measure must be used during the installation procedure to carefully monitor installation progress. The sand is slowly poured into the annulus between the riser pipe and temporary casing, as the casing is withdrawn. Sand should always be kept within the temporary casing during withdrawal in order to ensure an adequate sand pack. However, if too much sand is within the temporary casing (greater than 1 foot above the bottom of the casing) bridging between the temporary casing and riser pipe may occur. Centralizers may be used at the geologist's discretion, one above and one below the screen, to assure enough annular space for sand pack placement.

After the sand pack is installed to the desired depth (at least 1 foot above the top of the screen), then the bentonite pellet seal (or equivalent), can be installed in the same manner as the sand pack. At least 1 to 3 feet of bentonite pellets should be installed above the sand pack. Pellets should be added slowly and their fall monitored closely to ensure that bridging does not occur.

The cement-bentonite grout is then mixed and tremied into the annulus as the temporary casing or augers are withdrawn. Finally, the protective casing can be installed as detailed in Section 5.2.4.

5.3.2 Confining Layer Monitoring Wells

When drilling and installing a well in a confined aquifer, proper well installation techniques must be applied to avoid cross contamination between aquifers. Under most conditions, this can be accomplished by installing double-cased wells. This is accomplished by drilling a large-diameter boring through the upper aquifer, 1 to 5 feet into the underlying confining layer, and setting and pressure grouting or tremie grouting a large-diameter casing into the confining layer. The grout material must fill the space between the native material and the outer casing. A smaller diameter boring is then continued through the

confining layer for installation of the monitoring well as detailed for overburden monitoring wells. Sufficient time (determined by the field geologist), must be allowed for setting of the grout prior to drilling through the confined layer.

5.3.3 Bedrock Monitoring Wells

When installing bedrock monitoring wells, a large diameter boring is drilled through the overburden and approximately 5 –10 feet into bedrock. A casing (typically steel) is installed and either pressure grouted or tremie grouted in place. After the grout has cured, a smaller diameter boring is continued into bedrock to the desired depth. If the boring does not collapse, the well can be left open, and a screen is not necessary. If the boring collapses, then a screen is required and can be installed as detailed for overburden monitoring wells. If a screen is to be used, then the casing which is installed through the overburden and into the bedrock does not require grouting and can be removed when the final well installation is completed.

~~5.3.4 Drive Points~~

~~Drive points can be installed with either a sledge hammer, drop hammer, or a mechanical vibrator. The screen section is threaded and tightened onto the riser pipe with pipe wrenches. The drive point is simply pounded into the subsurface to the desired depth. If a heavy drop hammer is used, then a tripod and pulley setup is required to lift the hammer. Drive points typically cannot be manually driven to depths exceeding 10 feet.~~

~~Direct push sampling/monitoring point installation methods, using a direct push rig or drilling rig, are described in SOP SA-2.5.~~

~~5.3.5 Innovative Monitoring Well Installation Techniques~~

~~Certain innovative sampling devices have proven advantageous. These devices are essentially screened samplers installed in a borehole with only small-diameter tubes extending to the surface. This reduces drilling costs, decreases the volume of stagnant water, and provides a sampling system that minimizes cross-contamination from sampling equipment. Four manufacturers of these samplers include Timco Manufacturing Company, Inc., of Prairie du Sac, Wisconsin, BARCAD Systems, Inc., of Concord, Massachusetts, Westbay Instruments Ltd. of Vancouver, British Columbia, Canada and the University of Waterloo at Waterloo, Ontario, Canada.. Each manufacturer offers various construction materials.~~

5.4 Well Development Methods

The purpose of well development is to stabilize and increase the permeability of the gravel pack around the well screen, and to restore the permeability of the formation which may have been reduced by drilling operations. Wells are typically developed until all fine material and drilling water is removed from the well. Sequential measurements of pH, conductivity, turbidity, and temperature taken during development may yield information (stabilized values) regarding whether sufficient development has been performed. The selection of the well development method shall be made by the field geologist and is based on the drilling methods, well construction and installation details, and the characteristics of the formation that the well is screened in. The primary methods of well development are summarized below. A more detailed discussion may be found in Driscoll (1986).

5.4.1 Overpumping and Backwashing

Wells may be developed by alternatively drawing the water level down at a high rate (by pumping or bailing) and then reversing the flow direction (backwashing) so that water is passing from the well into the

formation. This back and forth movement of water through the well screen and gravel pack serves to remove fines from the formation immediately adjacent to the well, while preventing bridging (wedging) of sand grains. Backwashing can be accomplished by several methods, including pouring water into the well and then bailing, starting and stopping a pump intermittently to change water levels, or forcing water into the well under pressure through a water-tight fitting ("rawhiding"). Care should be taken when backwashing not to apply too much pressure, which could damage or destroy the well screen.

5.4.2 Surging with a Surge Plunger

A surge plunger (also called a surge block) is approximately the same diameter as the well casing and is aggressively moved up and down within the well to agitate the water, causing it to move in and out of the screens. This movement of water pulls fine materials into the well, where they may be removed by any of several methods, and prevents bridging of sand particles in the gravel pack. There are two basic types of surge plungers; solid and valved surge plungers. In formations with low yields, a valved surge plunger may be preferred, as solid plungers tend to force water out of the well at a greater rate than it will flow back in. Valved plungers are designed to produce a greater inflow than outflow of water during surging.

5.4.3 Compressed Air

Compressed air can be used to develop a well by either of two methods: backwashing or surging. Backwashing is done by forcing water out through the screens, using increasing air pressure inside a sealed well, then releasing the pressurized air to allow the water to flow back into the well. Care should be taken when using this method so that the water level does not drop below the top of the screen, thus introducing air into the formation and reducing well yield. Surging, or the "open well" method, consists of alternately releasing large volumes of air suddenly into an open well below the water level to produce a strong surge by virtue of the resistance of water head, friction, and inertia. Pumping of the well is subsequently done using the air lift method.

5.4.4 High Velocity Jetting

In the high velocity jetting method, water is forced at high velocities from a plunger-type device and through the well screen to loosen fine particles from the sand pack and surrounding formation. The jetting tool is slowly rotated and raised and lowered along the length of the well screen to develop the entire screened area. Jetting using a hose lowered into the well may also be effective. The fines washed into the screen during this process can then be bailed or pumped from the well.

6.0 RECORDS

A critical part of monitoring well installation is recording of all significant details and events in the site logbook or field notebook. The geologist must record the exact depths of significant hydrogeological features, screen placement, gravel pack placement, and bentonite placement.

A Monitoring Well Sheet (see Attachments to SOP SA-6.3) shall be completed, ensuring the uniform recording of data for each installation and rapid identification of missing information. Well depth, length, materials of construction, length and openings of screen, length and type of riser, and depth and type of all backfill materials shall be recorded. Additional information shall include location, installation date, problems encountered, water levels before and after well installation, cross-reference to the geologic boring log, and methods used during the installation and development process. Documentation is very important to prevent problems involving questionable sample validity. Somewhat different information will need to be recorded, depending on whether the well is completed in overburden (single- or double-cased), as a cased well in bedrock, or as an open hole in bedrock.

The quantities of sand, bentonite, and grout placed in the well are also important. The geologist shall calculate the annular space volume and have an idea of the quantity of material needed to fill the annular space. Volumes of backfill significantly higher than the calculated volume may indicate a problem such as a large cavity, while a smaller backfill volume may indicate a cave-in or bridging of the backfill materials. Any problems with rig operation or down-time shall be recorded and may affect the driller's final fee.

7.0 REFERENCES

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Barcelona, M. J., P. P. Gibb and R. A. Miller, 1983. A Guide to the selection of Materials for Monitoring Well Construction and Groundwater Sampling. ISWS Contract Report 327, Illinois State Water Survey, Champaign, Illinois.

U.S. EPA, 1980. Procedures Manual for Groundwater Monitoring of Solid Waste Disposal Facilities. Publication SW-611, Office of Solid Waste, U.S. EPA, Washington, D.C.

Driscoll, Fletcher G., 1986. Groundwater and Wells. Johnson Division, St. Paul, Minnesota, 1989.

ATTACHMENT A

RELATIVE COMPATIBILITY OF RIGID WELL CASING MATERIAL (PERCENT)

Potentially-Deteriorating Substance	Type of Casing Material						
	PVC 1	Galvanized Steel	Carbon Steel	Lo-carbon Steel	Stainless Steel 304	Stainless Steel 316	Teflon*
Buffered Weak Acid	100	56	51	59	97	100	100
Weak Acid	98	59	43	47	96	100	100
Mineral Acid/ High Solids Content	100	48	57	60	80	82	100
Aqueous/Organic Mixtures	64	69	73	73	98	100	100
Percent Overall Rating	91	58	56	59	93	96	100

Preliminary Ranking of Rigid Materials:

1 Teflon[7]
2 Stainless Steel 316
3. Stainless Steel 304
4 PVC 1
5 Lo-Carbon Steel
6 Galvanized Steel
7 Carbon Steel

* Trademark of DuPont

RELATIVE COMPATIBILITY OF SEMI-RIGID OR ELASTOMERIC MATERIALS (PERCENT)

Potentially-Deteriorating Substance	Type of Casing Material								
	PVC Flexible	PP	PE Conv.	PE Linear	PMM	Viton[7]*	Silicone	Neoprene	Teflon[7]*
Buffered Weak Acid	97	97	100	97	90	92	87	85	100
Weak Acid	92	90	94	96	78	78	75	75	100
Mineral Acid/ High Solids Content	100	100	100	100	95	100	78	82	100
Aqueous/Organic Mixtures	62	71	40	60	49	78	49	44	100
Percent Overall Rating	88	90	84	88	78	87	72	72	100

Preliminary Ranking of Semi-Rigid or Elastomeric Materials:

1 Teflon[7]
2 Polypropylene (PP)
3. PVC Flexible/PE Linear
4 Viton[7]
5 PE Conventional
6 Plexiglas/Lucite (PMM)
7 Silicone/Neoprene

* Trademark of DuPont

Source: Barcelona et al., 1983

ATTACHMENT B

COMPARISON OF STAINLESS STEEL AND PVC FOR MONITORING WELL CONSTRUCTION

Characteristic	Stainless Steel	PVC
Strength	Use in deep wells to prevent compression and closing of screen/riser.	Use when shear and compressive strength are not critical.
Weight	Relatively heavier.	Light-weight; floats in water.
Cost	Relatively expensive.	Relatively inexpensive.
Corrosivity	Deteriorates more rapidly in corrosive water.	Non-corrosive -- may deteriorate in presence of ketones, aromatics, alkyl sulfides, or some chlorinated hydrocarbons.
Ease of Use	Difficult to adjust size or length in the field.	Easy to handle and work with in the field.
Preparation for Use	Should be steam cleaned if organics will be subsequently sampled.	Never use glue fittings -- pipes should be threaded or pressure fitted. Should be steam cleaned when used for monitoring wells.
Interaction with Contaminants*	May sorb organic or inorganic substances when oxidized.	May sorb or release organic substances.

* See also Attachment A.

APPENDIX C

FIELD DOCUMENTATION FORMS

BORING LOG FOR: Tank Farm 3, CTO WE59
PROJECT NO.: 112G02710
LOGGED BY:
DRILLED BY (Company/Driller):
GRD. SURFACE ELEVATION:

TRANSCRIBED BY:
ELEVATION FROM:

BORING NO.:
START DATE:
COMPLETION: DATE:
MON. WELL NO.:
CHECKED BY:

DEPTH (FEET)	BLOWS PER 6"	SAMP REC. / SAMP LENG.	SAMPLING TIME & SAMPLE NO. (QA/QC STATUS)	DEPTH MAT'L CHG./ WELL PROF'L	SOIL DENSITY/ CONSIS. or ROCK HARD.	CLR	MATERIAL CLASSIFICATION	USCS or ROCK BRKN	REMARKS (moisture condition; odors; geological classification; rock weathering; etc.)	FIELD SCREENING DATA *METHOD* = [FID, (PPM)]

TYPE OF DRILLING RIG:
METHOD OF ADVANCING BORING:
METHOD OF SOIL SAMPLING:
METHOD OF ROCK CORING:
GROUNDWATER LEVELS:
OTHER OBSERVATIONS:

BORING NO.:

Tetra Tech NUS, Inc.

PAGE: 1 OF 1

TtNUS Form 0018

TETRA TECH NUS, INC.

PHOTOIONIZATION DETECTOR FIELD CALIBRATION LOG

Serial No.: ____________________

Model No.: ____________________

Decal No.: ____________________

Site Name/Location: Tank Farm 3 – NAVSTA Newport Newport, RI

Tetra Tech NUS Charge No.: 112G02710 / CTO WE59

CALIBRATION DATE	STANDARD GAS-ISOBUTYLENE	(AM) CALIBRATION READING Isobutylene Equiv. (ppm)	(PM) CALIBRATION CHECK Isobutylene Equiv. (ppm)	SIGNATURE	COMMENTS
	Lot # ____________ Conc. = __________ ppm	/		AM: PM:	
	Lot # ____________ Conc. = __________ ppm	/		AM: PM:	
	Lot # ____________ Conc. = __________ ppm	/		AM: PM:	
	Lot # ____________ Conc. = __________ ppm	/		AM: PM:	
	Lot # ____________ Conc. = __________ ppm	/		AM: PM:	
	Lot # ____________ Conc. = __________ ppm	/		AM: PM:	
	Lot # ____________ Conc. = __________ ppm	/		AM: PM:	
	Lot # ____________ Conc. = __________ ppm	/		AM: PM:	
	Lot # ____________ Conc. = __________ ppm	/		AM: PM:	

TtNUS Form 0006

TETRA TECH NUS, INC.

FIELD INSTRUMENT CALIBRATION LOG

SITE NAME: Tank Farm 3 NAVSTA Newport, Newport, RI

INSTRUMENT NAME LaMotte Turbidimeter MODEL No.:

SERIAL No.: DECAL No.: TETRA TECH NUS CHARGE No. 112G02710 / CTO WE59

DATE	INITIAL READING (AM)		PROCEDURE	FINAL READING (PM Cal Check)		SIGNATURE	COMMENTS
	0 NTU	10 NTU (span)	Per Manufacturer's Instruction	0 NTU	10 NTU (span)		
	/		Per Manufacturer's Instruction	/		AM: PM:	
	/		Per Manufacturer's Instruction	/		AM: PM:	
	/		Per Manufacturer's Instruction	/		AM: PM:	
	/		Per Manufacturer's Instruction	/		AM: PM:	
	/		Per Manufacturer's Instruction	/		AM: PM:	
	/		Per Manufacturer's Instruction	/		AM: PM:	
	/		Per Manufacturer's Instruction	/		AM: PM:	
	/		Per Manufacturer's Instruction	/		AM: PM:	
	/		Per Manufacturer's Instruction	/		AM: PM:	
	/		Per Manufacturer's Instruction	/		AM: PM:	
	/		Per Manufacturer's Instruction	/		AM: PM:	
	/		Per Manufacturer's Instruction	/		AM: PM:	
	/		Per Manufacturer's Instruction	/		AM: PM:	

Mod.TtNUS Form 0007

TETRA TECH NUS, INC.

FIELD MODIFICATION RECORD

Site Name: _____ Location: _____

Project Number: _____ Task Assignment: _____

To: _____ Location: _____ Date: _____

Description: _____

_____

_____

_____

Reason for Change: _____

_____

_____

Recommended Action: _____

_____

_____

Field Operations Leader (Signature): _____ Date: _____

Disposition/Action: _____

_____

_____

Project Manager (Signature): _____ Date: _____

Distribution:

		Others as Required:
Program Manager:	_____	
Project Manager:	_____	_____
Quality Assurance Officer:	_____	_____
Field Operations Leader:	_____	_____
Project File:	_____	_____

TtNUS Form 0003

TETRA TECH NUS, INC.

YSI MULTIPARAMETER WATER QUALITY METER CALIBRATION LOG

Site Name: TANK FARM 3

Serial No:

Job No: 112G02710

Model No:

Decal Letter:

Instrument is calibrated in accordance with manufactures instructions

Date		Conductivity					Dissolved Oxygen 100%			ORP		Solution	Barometric	
		1000 µS/cm	DI check	pH 4.0	pH 7.0	pH 10.0	% sat.	ppm	Cell Temp	mV	@ Temp	Temp ° C	Pressure, mm of Hg	Signature
	am													
	pm													
	notes:													
	am													
	pm													
	notes:													
	am													
	pm													
	notes:													
	am													
	pm													
	notes:													
	am													
	pm													
	notes:													
	am													
	pm													
	notes:													
	am													
	pm													
	notes:													

Calibration STD's

Lot Number:					NA		NA	NA
Expires:								

Tetra Tech NUS, Inc.

GROUNDWATER SAMPLE LOG SHEET

Project Site Name: Tank Farm 3
Project No.: 112G02710

[ ] Domestic Well Data
[X] Monitoring Well Data
[ ] Other Well Type: _____
[ ] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____
Type of Sample:
[ ] Low Concentration
[ ] High Concentration

SAMPLING DATA:

Date: / Time:	Color (Visual)	pH (S.U.)	S.C. (mS/cm)	Temp. (^{0}C)	Turbidity (NTU)	DO (mg/l)	Salinity (%)	Other
Method:								

PURGE DATA:

Date:	Volume	pH	S.C.	Temp.	Turbidity	DO	Salinity	Other
Method:								
Monitor Reading (ppm):								
Well Casing Diameter & Material Type:								
Total Well Depth (TD):								
Static Water Level (WL):								
One Casing Volume(gal/L):								
Start Purge (hrs):								
End Purge (hrs):								
Total Purge Time (min):								
Total Vol. Purged (gal/L):								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

TETRA TECH NUS, INC.

MONITORING WELL PURGE DATA SHEET – "LOW STRESS" GROUNDWATER

Site Name: Tank Farm 3 NAVSTA Newport, Newport, RI
Sample ID: _____.

Tetra Tech NUS Charge No. 112G02710 / CTO WE59 Page 1 of
QC: _____(If applicable)

Sample Method: Low Stress (flow) with Bladder Pump/Peristaltic pump
Depth Sampled: _____ ft bgs Screen Int. Depth _____ ft bgs
Sample Date & Time:___ /___ /2011 _____hours _____(Dup Time)
Sampler(s):
Data Recorded By:_____ Signature:
.
Well Diameter/Total Depth _____(ft below TOR); Stickup_____;
Visual Evidence of Sheen (Yes/No) _____;
Olfactory Evidence of Odor (Yes/No)
.

PID BZ= / W= / PW= PPM. Field Instrument Group A/B/C/D

Pre-tubing insertion WL _____ft btor; Post tubing insertion WL _____ft btor

Analyte	Preservative	Container requirements	Collected
			Yes / No
			Yes / No
			Yes / No
			Yes / No

Clock Time 24hr	Water Depth (ft below TOR)	Pump Dial 1	Purge Rate ml/min	Cum. Volume Purged Gals.	Temp °C	S. Cond. 2 µS/cm	DO mg/L	pH (S.U.)	ORP mV	Salinity (ppth)	Turbidity (NTU)	Comments

TtNUS Form 0009 (modified)

Well or Saturated Screen Volume (gallons)_____. 2in Screen Volume = 0.163 gal/ft or 616 ml per foot.
BZ=Breathing Zone, W=Well, PW=Purge Water

1. Pump dial setting (for example: hertz, cycle/min, etc.)
2. Siemens per cm (same as umhos/cm) at 25 °C.
3. Oxidation reduction potential (stand in for Eh).

TETRA TECH NUS, INC.

MONITORING WELL PURGE DATA SHEET – "LOW STRESS" GROUNDWATER

Site Name: Tank Farm 3 – RI NAVSTA Newport, Newport, RI
Sample ID: _____

Tetra Tech NUS Charge No. 112G02422 / CTO WE59 Page 2 of ___

Clock Time 24hr	Water Depth (feet below TOR)	Pump Dial 1	Purge Rate ml/min	Cum. Volume Purged Gals.	Temp °C	S. Cond. 2 µS/cm	DO mg/L	pH (S.U.)	ORP (mV)	Salinity (ppth)	Turbidity NTU	Comments

TtNUS Form 0009 (modified)

1. Pump dial setting (for example: hertz, cycle/min, etc.)
2. Siemens per cm (same as umhos/cm) at 25 °C.
3. Oxidation reduction potential (stand in for Eh).

Tetra Tech NUS, Inc.

QA SAMPLE LOG SHEET

Project Site Name: TANK FARM 3
Project Number: 112G02710
Sample Location: _____
QA Sample Type:

Sample ID Number: _____
Sampled By: _____
C.O.C. Number: _____

[ ] Trip Blank
[ ] Source Water Blank (Field Blank)
[ ] Rinsate Blank
[ ] Other Blank _____

SAMPLING DATA:

Date: _____
Time: _____
Method: Direct Pour

WATER SOURCE:

[ ] Laboratory Prepared
[ ] Purchased
[ ] Other _____
[ ] Tap
[ ] Fire Hydrant

PURCHASED WATER INFORMATION (If Applicable as Source or Rinsate Water):

Product Name: Reagent Grade Water (DIUF)
Supplier: _____
Manufacturer: _____
Order Number: _____
Lot Number: _____
Expiration Date: _____

RINSATE INFORMATION (If Applicable):

Media Type: _____
Equipment Used: _____
Equipment Type:
[ ] Dedicated
[ ] Reusable
[ ] Disposable

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected
VOC			YES / NO
EDB			YES / NO
PAH			YES / NO
Metals			YES / NO
Dioxins			YES / NO

OBSERVATIONS / NOTES:

Signature(s):

Tetra Tech NUS, Inc.

MONITORING WELL DEVELOPMENT RECORD

Well: ______________________________
Site: ______ Tank Farm 3
Date Installed: ______________________________
Date Developed: ______________________________
Dev. Method: ______________________________
Pump Type: ______________________________

Depth to Bottom (ft.): ____________________
Static Water Level Before (ft.): ____________
Static Water Level After (ft.): ____________
Screen Length (ft.): ____________________
Specific Capacity: ____________________
Casing ID (in.): ____________________

Responsible Personnel: ______________________________
Drilling Co.: ______________________________
Project Name: Data Gaps Assessment Tank Farm 3 CTO WE 59
Project Number: 112G02710-

Time	Estimated Purge Rate mL/min	Cumulative Water Volume (Gal.)	Water Level Readings (Ft. below TOC)	Temperature (Degrees C)	pH	Specific Conductance (Units _____)	Turbidity (NTU)	Remarks (odor, color, etc.)

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: _____ Tetra Tech NUS Charge No. _____

Sample ID: _____ QC Information: _____(if applicable)

Sample Method: _____

Depth Sampled: _____feet

Sample Date & Time: ____/____/_____ _____hours Dup_____hours

Sampler(s): _____

_____

_____

Data Recorded By: _____

Signature

PID/OVA Monitor Reading: _____ ppm

TYPE OF SAMPLE: (Check all that apply)

_____ Soil
_____ Sediment
_____ Lagoon/Pond
_____ Grab
_____ Trip Blank*
_____ Rinsate Blank*
_____ Field Duplicate collected
_____ Other (Specify): _____

_____

_____

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.)

_____

_____

_____

SAMPLE DATA/REMARKS: _____

_____

_____

ANALYSIS	BOTTLE LOT No.	NOTES/SKETCH:

TtNUS Form 0005

TETRA TECH NUS, INC.

VOC SOIL SAMPLE COLLECTION/PRESERVATION LOG SHEET

Sample Container Lot # _____ Preserved in the field Date: _____

			LOW CONCENTRATION VOC				HIGH CONCENTRATION				LAB
Sample #	Bottle Letter	Vial ID Number	Volatile Free Water	Tare Weight Vial + 5 ml H_2O (g)	Final Weight Vial + H_2O + Sample (g)	Soil Sample Weight (g)	Methanol (mL)	Tare Weight Vial + Pres. (g)	Final Weight (g)	Soil Sample Weight (g)	Final Weight Vial + Pres. + Sample (g)

TtNUS Form 0040

Note: Tare weight = vial, preservative, cap and magnetic stirrer, if applicable

Laboratory Analysts Initials: _____ Date: _____

TETRA TECH NUS, INC.

WEEKLY FIELD SUMMARY REPORT

TO: _____ FROM: _____

SITE: _____ JOB NO.: _____

DATES: _____ TO _____

DAY/DATE: _____
WEATHER: _____

PERSONNEL ONSITE: _____

SITE ACTIVITIES: _____

DAY/DATE: _____ WEATHER: _____

PERSONNEL ONSITE: _____

SITE ACTIVITIES: _____

DAY/DATE: _____ WEATHER: _____

PERSONNEL ONSITE: _____

SITE ACTIVITIES: _____

DAY/DATE: _____ WEATHER: _____

WEEKLY FIELD SUMMARY REPORT (continued)

PERSONNEL ONSITE: _____

SITE ACTIVITIES: _____

DAY/DATE: _____ WEATHER: _____

PERSONNEL ONSITE: _____

SITE ACTIVITIES: _____

DAY/DATE: _____ WEATHER: _____

PERSONNEL ONSITE: _____

SITE ACTIVITIES: _____

DAY/DATE: _____ WEATHER: _____

PERSONNEL ONSITE: _____

SITE ACTIVITIES: _____

TETRA TECH NUS, INC.

WELL INSPECTION AND GROUNDWATER LEVEL MEASUREMENT SHEET

WELL NUMBER: _____ PROJECT NAME: _____

DATE/TIME: _____ PROJECT MANAGER: _____

INSPECTED BY: _____

_____

VENT WELL

MONITORING INSTRUMENT READING: _____

LEL/02 READING: _____

WELL INSPECTION/GROUNDWATER LEVEL MEASUREMENT

WELL DEPTH (FEET FROM TOP OF PVC) _____

WATER LEVEL DEPTH (FEET FROM TOP OF PVC) _____

WELL STICK-UP _____

CASING STICK-UP (FEET) _____

WELL DIAMETER (INCHES) _____

WELL CONSTRUCTION (PVC, STEEL, ETC.) _____

LOCKED UPON ARRIVAL?	YES	NO
LOCKED REPLACED?	YES	NO
OBSTRUCTIONS?	YES	NO
WELL RELABELED?	YES	NO
SLUG TEST CONDUCTED?	YES	NO (If YES, refer to "Hydraulic Conductivity Testing Data Sheet")

GENERAL CONDITION/COMMENTS: _____

_____

_____

_____

_____

TETRA TECH NUS, INC.

RECORD OF REVIEW

1. TITLE AS IT APPEARS ON THE DOCUMENT -- (See Instruction #1 on reverse for definition of Documents):

_____

_____

2. DATE OF DOCUMENT: _____ DOCUMENT CONTROL No.: _____

AUTHORS: _____ TtNUS JOB CHARGE No.: _____

TYPE (DRAFT, DRAFT FINAL, FINAL): _____ SUBMITTAL DATE: _____

3. REVIEWER AND STATUS REVIEWERS ASSIGNED BY: _____ Date _____

Assigned Reviewer (See Instruction #3)	Disapproval		Approved with Suggestions		Reviewer Requires Final Verification		Approved By	
	(Initials)	(Date)	(Initials)	(Date)	(Initials)	(Date)	(Initials)	(Date)
a)								
b)								
c)								
d)								

4. COMMENTS (Explain conditions and comments, or state where comments and edits are provided. Attach additional pages as needed):

5. AUTHOR COMMENTS/RESOLUTION:

6. APPROVAL FOR TRANSMITTAL:

_____ DATE: _____

See #7 on Reverse for Authority to Approve Transmittals

TtNUS Form 0001 (Revised 3/12/02)

c: File No. _____ - _____ (see #8 on Reverse for Correct File Designation)

INSTRUCTIONS FOR COMPLETION OF RECORD OF REVIEW FORM

1) Record of Review (ROR) forms document compliance with quality control procedures. The ROR can be used to document review of **reports, technical memoranda, drawings, financial and cost estimates, contract documents, tables of data or other information calculations, etc**. Collectively, all these items are considered "documents" for the purposes of the Record of Review.

2) The author(s) complete Sections 1 and 2 and submits the form to the Project Manager. All items in Sections 1 and 2 should be completed. Section 1 states the exact title of the document. Any item in these sections that is not applicable should be indicated as "N/A".

3) The reviewers are assigned by the responsible authority designated in the contract and/or contract procedures, guidance, QA/QC Plans. If no authority is designated, then the Project Manager assigns the reviewers. The person who assigns the reviewers, and the reviewers names, are entered into Section 3 of the form. If more than four reviewers are assigned, use additional forms. Check with appropriate Project Manager, or Program Manager to determine who is the authorized person to assign reviewers. For EPA RAC I, the Deputy Program Manager or Program Manager assigns the reviewers.

4) The author(s) attach the form to the document to be reviewed and submit the review packages to the reviewers. (For expediting purposes, copies of the review package may be made and circulated for parallel review. Each review package should have a copy of the ROR attached.)

5) Reviewer(s) perform the review, make comments, and initial and date the appropriate status column in Section 3 of the review form. Minor comments may be made in Section 4 of the form, additional sheets may be used, or comment may be made directly in the text of the document, and note in Section 4 that comments are in the draft document. Definition and actions for the Reviewers conditions in Section 3 are as follows:

   - Disapproval - Reviewer has serious problems with the document, and document should not be issued without major changes. Typically, these problems include perceived errors in fact or interpretation, incorrect assumptions, technical conclusions that are not supported by the facts, errors in data or calculations, incompleteness, or poor technical writing. Place your initials in the Disapproval column and fill in disapproval date. **Since this category should be used for serious problems, the reviewer should immediately discuss the issues with the author(s) for resolution,** in addition to completing the form.

   - Approved with Suggestions - Reviewer approves the document providing comment and conditions. Typically, this category includes edits for clarification, suggested rewordings, suggested additions or deletions to enhance the documents technical presentation or completeness. Suggestions are cited in Section 4, on separate attached sheets, or in the review copy of the document.

   - Reviewer Requires Final Verification – The Reviewer must check the corrected document to verify that the issues raised in a "Disapproval" or "Approved with Suggestions" evaluation, and needs to verify consideration of reviewers comments in the document. Authors will return the reviewer's comments with the revised document to verify comments were addressed.

   - Approved By - Initiated by the reviewer signifying approval of the document. Reviewers who initially "Disapproved" the document, and/or "Required Final Verification do not initial and date this column until the document is revised to their satisfaction, or the appropriated authority has resolved the reviewers issues.

6) Author resolves comments and explains briefly resolutions of the reviewer's conditions, and briefly describes how the issues were resolved in Section 5. The author must include a brief description of actions taken. In case of disagreements, the project manager, or program manager is responsible for arbitration. Arbitration resolution should also be explained in Section 5.

7) "Approval for Transmittal" must be obtained prior to transmittal. The approving authority is project and contract specific. "Approval for Transmittal" only acknowledges that the appropriate review procedures have been implemented. For EPA RAC contracts, the approving authority is the Program or Deputy Program Manager, or the Quality Assurance Manager. Check with the project or program manager to determine who the approving authority for transmittal is for other projects.

8) A copy of the review form should be filed along with the document. The original record of review form should be filed in the following file sections:

RAC	Program File -0310	Wilmington Overhead 1052-0520	
Navy	Project File XXXX-2.7	Miscellaneous Projects	XXXX-5.3

Tetra Tech NUS, Inc.

QA SAMPLE LOG SHEET

Project Site Name: _____ Sample ID Number: _____
Project Number: _____ Sampled By: _____
Sample Location: _____ C.O.C. Number: _____
QA Sample Type:

[] Trip Blank [] Rinsate Blank
[] Source Water Blank [] Other Blank _____

SAMPLING DATA:

Date: _____
Time: _____
Method: _____

WATER SOURCE:

[] Laboratory Prepared [] Tap
[] Purchased [] Fire Hydrant
[] Other _____

PURCHASED WATER INFORMATION (If Applicable as Source or Rinsate Water):

Product Name: _____
Supplier: _____
Manufacturer: _____
Order Number: _____
Lot Number: _____
Expiration Date: _____

RINSATE INFORMATION (If Applicable):

Media Type: _____
Equipment Used: _____
Equipment Type:
[] Dedicated
[] Reusable

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected
Volatiles	Cool 4°C & HCl		YES / NO
Semivolatiles	Cool 4°C		YES / NO
Pesticide / PCB	Cool 4°C		YES / NO
Metals	Cool 4°C & HNO_3		YES / NO
Cyanide	Cool 4°C & NaOH		YES / NO

OBSERVATIONS / NOTES:

Signature(s):

TETRA TECH NUS, INC.

SAMPLE LOG SHEET - SOLID PHASE

Site Name: _____ Tetra Tech NUS Charge No. _____

Sample ID: _____ QC Information: _____(if applicable)

Sample Method: _____

Depth Sampled: _____feet

Sample Date & Time: ____/____/_____ _____hours Dup_____hours

Sampler(s): _____

_____

_____

Data Recorded By: _____

Signature

PID/OVA Monitor Reading: _____ ppm

TYPE OF SAMPLE: (Check all that apply)

_____ Soil
_____ Sediment
_____ Lagoon/Pond
_____ Grab
_____ Trip Blank*
_____ Rinsate Blank*
_____ Field Duplicate collected
_____ Other (Specify): _____

_____

_____

Description: (Sand, Clay, Muck, Peat, Dry, Moist, Wet, Etc.)

_____

_____

_____

SAMPLE DATA/REMARKS: _____

_____

_____

ANALYSIS	BOTTLE LOT No.	NOTES/SKETCH:

TtNUS Form 0005

APPENDIX D

PROJECT-SPECIFIC FIELD TASK PROCEDURES

APPENDIX D

PROJECT-SPECIFIC SAMPLING PROCEDURES

Soil, groundwater and sediment sampling, and well development, will be performed at Tank Farm 3 according to the SOPs listed in Worksheet # 21.

Quality control (QC) samples will be collected as part of the investigation, including field duplicates, rinsate blanks, and trip blanks. Samples will also be assigned on the chain-of-custody (COC) form for laboratory QC analyses. Worksheet #20 summarizes the QC samples to be collected for each matrix.

The sample locations are presented on Figures 3 through 5 and on Worksheet #18. Worksheet #18 also presents the analytical groups for each sample location. Worksheet #19 presents the analytical methods, sample container types, preservative requirements, and the maximum holding times for the associated analyses.

Samples will be identified in accordance with the sample location identification system presented in Worksheet #27. All laboratory analytical samples will be kept on ice in coolers and will be shipped with appropriate QC samples. Samples will be handled and delivered in accordance with the COC procedures detailed in Worksheet #27.

1.0 SOIL SAMPLING PROCEDURES

For this DGA soil borings will be advanced at three AOCs (AOC-001, AOC-020 and Building 227) within Tank Farm 3 at the locations depicted on Figures 3 through 5. At each AOC soil borings will be advanced for continuous soil sampling to a depth of 10 feet below ground surface, or bedrock. A variety of drilling techniques may be used depending on the conditions encountered at the site. Direct push technology, hollow-stem auger and/or drive and wash drilling techniques will be implemented.

Drilling will be performed by a subcontractor according to the procedures in SOP SA-2.5, for direct push technology and/or SOP and/or GH-1.3 for Soil and Rock Drilling Methods. All down-hole drilling equipment will be steam-cleaned before use at each boring. Continuous soil samples will be retrieved at 2-foot intervals throughout the length of the boring using a 2-inch ID, stainless steel split-barrel sampler for conventional drilling techniques. For direct-push, a 2-inch ID, 4 or 5-foot long, stainless steel, macro-core sampler with an inner acetate liner will be used to collect the soil samples. All required information will be recorded on the boring log, in accordance with SOP GH-1.5 and the procedures described below.

Soil samples will be collected for laboratory analysis from the soil cores in accordance with SOP SA-1.3 using the procedures described below. From every soil boring one surface soil sample (0-1 foot) and one subsurface soil sample will be collected for laboratory analysis. The depth intervals for subsurface soil samples at AOC-020 and Building 227 will be determined as described in Worksheet #17. The depth intervals for subsurface soil samples at AOC-001 will be determined as described below. At AOC-001, soil samples will be analyzed for extractable total petroleum hydrocarbons (ExTPH), gasoline range organics (GROs), VOCs, PAHs, dioxins and metals. At AOC-020, soil samples will be analyzed for PCBs. At Building 227 soil samples will be analyzed for ExTPH, GRO, PCBs and metals. (See Worksheet #18 for details.) The boring log will act as the sample log sheet for samples collected.

For each continuous 2-foot interval, the split-barrel sampler or acetate liner will be opened, visually inspected, and scanned with a photo-ionization detector (PID) portable monitoring instrument. At AOC-001 and B227 borings, a grab sample first will be collected from the most heavily contaminated portion of the surface soil interval sample (0-1 foot bgs) and each 2-foot subsurface soil interval sample (2-10 feet bgs), based on the initial screening results and/or visual and olfactory observations. From each grab sample, the following aliquots will be collected:

- One grab sample aliquot will be collected according to the sampling procedures described below for volatile samples and stored for laboratory VOC and/or GRO analysis (surface soil interval) or stored temporarily for possible laboratory VOC and/or GRO analysis (subsurface soil intervals).
- One grab sample aliquot will be collected for jar-headspace total VOC analysis, performed in the field.
- One grab sample aliquot will be collected for determination of percent moisture (see procedures below).

The remainder of each soil interval sample will be kept for possible laboratory analysis for the non-volatile parameters. (The 1-2 feet bgs soil interval will be discarded unless the initial screening results or observations indicate that a grab sample should be collected. If a grab sample is collected, then the subsequent continuous 2-foot intervals will begin at 1-foot bgs.) After all the soil cores have been recovered from a borehole and grab sample aliquots collected, a subsurface soil 2-foot interval sample will be selected for laboratory analysis based on the jar headspace screening results and/or visual and olfactory observations.

At AOC-001 and B227 boreholes, the remainder of the soil from the 1-foot surface soil or selected 2-foot subsurface soil sample interval will be uniformly hand mixed to form a composite sample and split into aliquots for the remaining non-volatile analyses (DRO, PAHs, metals, and dioxins).

At AOC-020 borings, the entire contents of each 1-foot surface soil or selected 2-foot subsurface soil sample interval will be similarly mixed and split into aliquots for PCB analysis. For these aliquots, the sampling procedures below for non-volatile parameters will be followed.

Soil Sampling Procedures for Volatile Laboratory Samples (Grab)

Each soil sample for possible volatile analysis (VOCs and GRO) will be collected using a cut syringe or equivalent device. VOC aliquots will be placed in two sodium bisulfate ($NaHSO_4$)-preserved vials with septa caps and one methanol-preserved vial with a septa cap, according to the SW-846 Method 5035A (July 2002). GRO aliquots will be placed in two methanol-preserved vial with a septa cap. The vials will be maintained at ≤ 6 °C for up to 14 days. The following procedures should be followed for the soil volatile sample collection:

1. Label two pre-tare weighed 40-mL amber vials containing 1 g sodium bisulfate in 5 mL of reagent water, and one 40-mL amber vial containing 5 mL of methanol for the VOC aliquots with the sample location number and a bottle letter such as A, B, etc.

2. Label two pre-tare weighed 40-mL amber vials containing 5 mL of methanol for the GRO aliquots with the sample location number and a bottle letter such as A, B, etc.

3. Collect approximately 5 grams of sample by coring or stabbing the soil with a 10-mL pre-cut syringe. Extrude the sample into one of the 40-mL vials containing 5 mL of preservative (bisulfate in reagent water, or methanol). The soil must be immersed in the preservative; recollect the sample using a smaller volume if necessary. Avoid touching the threads on the vial's neck and avoid loss of preservative by evaporation. Cap the vial and invert it several times to mix the sample.

4. Weigh each sample vial to the nearest 0.01 gram and record the weight on the field log sheet. Repeat the sample collection procedure for the remaining vials. Pack and ship to the laboratory. Include the field log sheet containing the sample weight information with the samples.

5. If non-aqueous phase liquids (NAPL) is noted within the soils, a reduced volume of approximately 1 to 2 grams should be collected as a separate "medium concentration" (NAPL) sample.

Quality assurance and quality control samples will also be collected, see Worksheet #20 for details. Following the collection of the first set of volatile containers, collect the field duplicate from the same sampling interval.

Every effort should be made to obtain the percent moisture soil aliquot (see below) as close as possible to the location where the volatile sample was collected.

Soil Sample for Percent Moisture

Fill one 2-oz container with soil representing the same locations where the 40-mL volatile vial samples were collected. Every effort should be made to obtain the percent moisture soil aliquot as close as possible to the location where the volatile samples were collected.

Soil Sampling Procedures for Non-VOC Parameters

1. Record all required data on the boring log, which will also serve as the soil sample log sheet. Include the sampling equipment, sampling personnel, date, time, depth of sample, and sample analyses. Use the boring log to record soil descriptions, depth of strata changes, and sample depth intervals. Classify the soil sample visually using the Unified Soil Classification System (ASTM D-2488-98).

2. Label appropriate sample jars with the sample location number, sampler's name, date, and analytical fractions.

3. Transfer the soil from the sampler to a decontaminated stainless-steel bowl using only decontaminated stainless steel trowels, and homogenize the sample.

4. If there is insufficient sample volume to fill all the containers for analyses, an equal amount of material from the intervals immediately above and below the selected sample interval may be used to supplement the composite sample to ensure sufficient sample quantity for all analyses. Alternatively, a second boring immediately adjacent to the original boring could be advanced to the desired depth to obtain additional sample volume. Document the method used in the boring log.

5. Remove any large particles such as twigs, gravel or artificial fill too large to be sent for analysis. Document the removal of material on the boring log.

6. Fill the appropriate sample containers with the soil sample.

7. For field duplicate samples, after homogenization, fill one set of sample containers for the original sample and fill another set for the field duplicate sample.

8. Ensure that the samples are properly labeled, maintained in coolers with ice, and that the COC procedures described in Worksheet 27 are followed. Package and ship the sample coolers to the appropriate laboratory for overnight delivery.

9. Decontaminate the sampling equipment before reuse.

Care should be taken in handling all soil samples to ensure that the exterior of the sample containers are clean and free of soils before shipping to the laboratory.

2.0 MONITORING WELL DEVELOPMENT

Each monitoring well that is scheduled for sampling will be redeveloped before sampling occurs. Wells will be developed using a pump and surge method with a Waterra pump and foot valve, until the turbidity reaches 10 Nephelometric Turbidity Units (NTUs) or 2 hours of well development elapses (whichever occurs first). Any well that dries up during development, even at lower pumping rates, will be evacuated dry a minimum of 3 times, after a 90% recharge, to consider development complete. A minimum of 1 week shall elapse after well development before collecting groundwater samples.

3.0 GROUNDWATER SAMPLING

A total of four groundwater samples will be collected from Tank Farm 3, one at each AOC.

- GZ-301 (AOC-001) will be sampled for VOCs, EDB, PAHs, and metals,
- GZ-314 (AOC-020) will be sampled for PCBs,
- Lastly, GZ-318 and a new shallow monitoring well installation (TF3-ECH-MW01), located in the electrical control house area (Building 227) will be sampled for PCBs and metals.

The groundwater samples will be collected following EPA's "low flow sampling protocol" (SOP GW 0001). This protocol will be modified for collection of groundwater samples from saturated well screens greater than 10 feet (described below). This method emphasizes the need to minimize water-level drawdown and groundwater pumping rates in order to collect samples with minimal alterations to groundwater chemistry.

Water level measurements will be obtained using an electronic water level indicator with a weighted cord that is accurate to 0.01 feet. In addition, an oil/water interface probe will be used to identify the potential presence of non-aqueous phase liquid (NAPL) in each well. If NAPL is present, its thickness in the well should also be measured using the probe. Water level measurements in wells will be recorded from the surveyed measuring point at the top of the inner well casing. The electronic water level indicator will be decontaminated between each well, following the procedure out lined in SOP SA 7.1 (Appendix A). If NAPL is present in a sufficient amount it will be sampled following the procedures outlined in Section 4 of this Appendix (E.2).

Submersible bladder pumps with polyethylene bladders will be used for low-flow groundwater sampling. Dedicated pumps may be used; non-dedicated pumps will be decontaminated between each well in accordance with this UFP SAP and SOP SA 7.1. The pumping system will be designed to be consistent with the intent of the low-flow groundwater sampling method. Dedicated tubing will be used for each monitoring well to minimize cross-contamination between monitoring wells.

During well purging, water level drawdown, flow rate, and water quality parameters will be recorded on groundwater collection forms. Groundwater will be pumped through a flow-through cell and the pH, conductivity, temperature, dissolved oxygen (DO), and oxidation-reduction potential (ORP) will be measured with a water-quality instrument. The instrument will be calibrated as described in Worksheet #22, in accordance with manufactures instructions. Turbidity will be measured separately with a nephelometer. Every effort will be made to lower the turbidity to less than 5 Nephelometric Turbidity Units (NTUs) before sampling. If turbidity below 5 NTU cannot be achieved, samples will still be collected if all parameters are stable and the 2-hour purging limit is reached. Purging is considered complete and sampling may begin when all parameters have stabilized. Stabilization is considered to be achieved when three consecutive readings, taken at 3- to 5-minute intervals, are within the following limits:

- Turbidity (+ or – 10% if greater than 5 NTU);
- DO (10% for values greater than 2 mg/L; 0.5 mg/L for values less than 2 mg/L);
- Specific conductance (3%);
- Temperature (3%);
- pH (+ or - 0.5 units);
- ORP (+ or - 10 millivolts); and
- Drawdown (no more than 0.3 feet).

EPA Region 1 low flow sampling procedure is modified for monitoring wells with saturated screen lengths greater than 10-ft. The modifications are described below.

Saturated screen lengths greater than 10 feet but not more than 20 feet:

The purging and sampling of monitoring wells with saturated screen lengths greater than 10 but not more than 20 feet will be performed using the following modifications:

1. Place the tubing about 1 foot below the top of the screen, or about 1 foot below the static water level if the screen is not fully saturated.
2. Pump well at a low flow rate to minimize drawdown (goal: not more than 0.3 feet).
3. Extract one saturated screen volume including drawdown volume.
4. Begin monitoring stabilization parameters and continue purging until stabilization criteria are met
5. Collect groundwater samples.

After 2 hours if the purging/stabilization process is not complete, the FOL will consult with the Project Manager who will make the determination to continue or to collect the groundwater sample at that time. If the purging/stabilization process is not complete (after a minimum of 2 hours) prior to sampling, the data from that sample will be qualified with information regarding which parameters did not meet the stabilization criteria. The reason for terminating the sampling at 2 hours will be noted on the sample collection form.

Monitoring wells that have saturated well screen lengths greater than 20 feet

The purging and sampling of monitoring wells that have saturated well screen lengths greater than 20 feet will be performed using the following modifications:

1. Place the tubing about 1 foot below the top of the screen, or about 1 foot below the static water level if the screen is not fully saturated.
2. Remove three well saturated screen volumes including drawdown volume. During purging adjust pumping rates to maintain water level above the top of the screen (if static water level is above top of screen).
3. Begin monitoring stabilization parameters and continue purging until stabilization criteria are met.
4. Collect groundwater sample.

After 2 hours if the purging/stabilization process is not complete, the FOL will consult with the Project Manager who will make the determination to continue or to collect the groundwater sample at that time. If the purging/stabilization process is not complete (after a minimum of 2 hours) prior to sampling, the data from that sample will be qualified with information regarding which parameters did not meet the

stabilization criteria. The reason for terminating the sampling at 2 hours will be noted on the sample collection form.

If difficulties arise during low stress (low flow) sample collection procedures (i.e. minimum drawdown is not obtainable, it can be documented that the well is not yielding fresh water despite the drawdown, or water chemistry readings do not show a stabilization pattern), affected wells may be sampled using a bailer or a peristaltic pump. In the event that the recovery time of the well is very slow (e.g., 24 hours), sample collection can be delayed until the following day. If the well is incapable of producing a sufficient volume of sample at any time, obtain the largest sample quantity available and record the quantity in the logbook. Any and all problems encountered during purging and sampling will be recorded in the site logbook. Sufficient information will be documented to support all field decisions. Deviations will be discussed with the Project Manager to determine the appropriate next step. Any significant deviation from the sampling protocol shall be proposed in a Field Modification Record.

If turbidity is below 10 NTUs, then one groundwater sample will be collected for metals analysis and that sample will represent total metals concentrations. If turbidity cannot be reduced to below 10 NTUs, following the low-flow stabilization protocol, then two samples for metals analysis will be collected: one for total metals analysis (unfiltered), and the other sample field filtered (0.45 micron pore size) for dissolved metals concentrations.

Groundwater Sample Collection for Laboratory Analysis

Groundwater samples for laboratory analyses must be collected before the water has passed through the flow-through cell; therefore, the discharge tubing will be disconnected from the flow-through cell and the in-line device will be used to directly collect aliquots for sample analyses.

Sample containers will be filled in order of decreasing volatility: VOCs, EDB, PAHs and metals. Care should be taken in handling all groundwater samples to ensure that the exterior of the sample containers are clean and free of liquids before shipping.

4.0 SEDIMENT SAMPLING

Final sample locations will be determined in the field in order to target depositional areas, or areas with visual evidence of contamination.

Sediment samples will be collected in a downstream to upstream order, so as to not disturb downstream locations before they are sampled. Samples will be collected using a decontaminated stainless steel

hand auger. Sediment samples will be analyzed for VOCs, GRO, ExTPH, PAHs, dioxins and metals, and samples will not be collected during, or within 24 hours of a major rain event.

Samples for VOC and GRO analysis will be collected directly from the hand auger following the procedures described above in Soil Sampling for Volatile Laboratory Samples. Once sediment for VOC and GRO analysis has been collected the remaining sediment in the auger bucket will be transferred to a stainless steel pan and homogenized; sediment will then be sampled following the procedure described above in the Soil Sampling Procedure for Non-Volatile Laboratory Samples. The appropriate sample jars will be filled and labeled, and then placed on ice.

5.0 MONITORING WELL CONSTRUCTION AND DEVELOPMENT

One new groundwater well, will be installed according to the procedures in SOP GH-2.8. The borings will be advanced using drilling techniques, as specified above. Continuous split spoon samples will be collected during advancement in order to characterize soils.

The newly installed well will consist of a 2-inch ID Schedule 40 PVC equipped with a minimum 10-foot long well screen, (typically 0.010 to 0.020 inch slot openings), surrounded by 20-40 grade silica sand, and a bottom plug. A 5-foot long well screen is acceptable if the geologic unit thickness is less than 10 feet. All connections will be flush-treaded. Silica sand shall be placed 2 feet above the top of the well screen, followed by a minimum of 2 feet of bentonite rock chips hydrated in place. Cement/ bentonite grout will fill the remainder of the annular space to approximately 3.5 feet bgs. A 6-inch sand drainage layer shall be placed on top of the grout. A steel protective casing (4-inch diameter by 5-ft long) will be centered around the well. Quikrete™ (or equivalent) will be used to fill the annular space above the sand drainage layer and form a pad around the well to a minimum radius of 1 foot. The top of the well pad shall be flush with ground surface. Silica sand shall be placed between the well and the protective casing. The protective casing will be painted in a rust-preventive paint and secured with a padlock.

The new monitoring well will be developed no sooner than 48 hours after well completion. Any existing wells that are scheduled to be sampled and have not been developed within the last twelve months will be re-developed before sampling. Wells will be developed using a pump and surge method with a Waterra™ inertial pump and foot valve, until the turbidity reaches 10 NTUs or 2 hours of well development elapses (whichever occurs first). Any well that dries up during development, even at lower pumping rates, will be evacuated dry a minimum of 3 times, after a 90% recharge, to consider development complete. A minimum of 1 week shall elapse after well development before collecting groundwater samples.

APPENDIX E

LABORATORY CERTIFICATION AND SOPs

LABORATORY ACCREDITATION BUREAU®

Certificate of Accreditation

ISO/IEC 17025:2005 Certificate Number L2223

Katahdin Analytical Services, Inc.

600 Technology Way
Scarborough ME 04074

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation valid through: February 1, 2016

R. Douglas Leonard, Jr., President, COO
Laboratory Accreditation Bureau
Presented the 1st of February 2013

*See the laboratory's Scope of Accreditation for details of accredited parameters
**Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).

LABORATORY ACCREDITATION BUREAU

Certificate # L2223

Scope of Accreditation
For
Katahdin Analytical Services, Inc.

600 Technology Way
Scarborough, ME 04074
Leslie Dimond
207-874-2400

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.2) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to Katahdin Analytical Services to perform the following tests:

Accreditation granted through: February 1, 2016

Testing - Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	2, 4`-DDD
GC/ECD	EPA 8081B	2, 4`-DDE
GC/ECD	EPA 8081B	2, 4`-DDT
GC/ECD	EPA 608; EPA 8081B	4, 4`-DDD
GC/ECD	EPA 608; EPA 8081B	4, 4`-DDE
GC/ECD	EPA 608; EPA 8081B	4, 4`-DDT
GC/ECD	EPA 608; EPA 8081B	Aldrin
GC/ECD	EPA 608; EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Alpha-Chlordane
GC/ECD	EPA 608; EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Cis-Nonaclor
GC/ECD	EPA 608; EPA 8081B	Chlordane (tech.)
GC/ECD	EPA 608; EPA 8081B	delta-BHC
GC/ECD	EPA 608; EPA 8081B	Dieldrin
GC/ECD	EPA 608; EPA 8081B	Endosulfan I
GC/ECD	EPA 608; EPA 8081B	Endosulfan II

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 608; EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 608; EPA 8081B	Endrin
GC/ECD	EPA 608; EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin Ketone
GC/ECD	EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	gamma-Chlordane
GC/ECD	EPA 608; EPA 8081B	Heptachlor
GC/ECD	EPA 608; EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Hexachlorobenzene
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Mirex
GC/ECD	EPA 8081B	Oxychlordane
GC/ECD	EPA 608; EPA 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 8081B	trans-Nonachlor
GC/ECD	EPA 608; EPA 8082A	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 608; EPA 8082A	Aroclor-1260 (PCB-1260)
GC/ECD	EPA 8082A MOD	Aroclor-1262 (PCB-1262)
GC/ECD	EPA 8082A MOD	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl (BZ 206)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5,6-Octachlorobiphenyl (BZ 195)
GC/ECD	EPA 8082A	2,2',3,3',4,4',5-Heptachlorobiphenyl (BZ 170)
GC/ECD	EPA 8082A	2,2',3,3',4,4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5', 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5-Hexachlorobiphenyl (BZ 138)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 6, 6'-Heptachlorobiphenyl (BZ 184)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A	2, 2', 3, 4', 5, 5', 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	EPA 8082A	2, 2', 3, 4, 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	EPA 8082A	2, 2', 3, 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	EPA 8082A	2, 2', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	EPA 8082A	2, 2', 4, 5, 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	EPA 8082A	2, 2', 4, 5-Tetrachlorobiphenyl (BZ 48)
GC/ECD	EPA 8082A	2, 2', 4, 5'-Tetrachlorobiphenyl (BZ 49)
GC/ECD	EPA 8082A	2, 2', 5, 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	EPA 8082A	2, 2', 5-Trichlorobiphenyl (BZ 18)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 189)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	EPA 8082A	2, 3', 4, 4',5'-Pentachlorobiphenyl (BZ 123)
GC/ECD	EPA 8082A	2, 3', 4, 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	EPA 8082A	2, 3, 4, 4', 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	EPA 8082A	2, 4, 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	EPA 8082A	2, 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 126)
GC/ECD	EPA 8082A	3, 3', 4, 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	EPA 8082A	3, 4, 4', 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	EPA 8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	EPA 8151A	2, 4, 5-T
GC/ECD	EPA 8151A	2, 4-D
GC/ECD	EPA 8151A	2, 4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Silvex (2, 4, 5-TP)
GC/FID	EPA 8015C/D MOD	Diesel range organics (DRO)
GC/FID	EPA 8015C/D MOD	Total Petroleum Hydrocarbon (TPH)
GC/FID	EPA 8015C/D MOD	Gasoline range organics (GRO)
GC/FID/PID	MA DEP VPH	Volatile Organic Hydrocarbons
GC/FID	MA DEP EPH	Extractable Petroleum Hydrocarbons
GC/FID	CT ETPH	Total Petroleum Hydrocarbons
GC/FID	TNRCC Method 1005	Total Petroleum Hydrocarbons
GC/FID	FL-PRO	Petroleum Range Organics
GC/ECD	EPA 8011; EPA 504	1, 2-Dibromoethane (EDB)
GC/ECD	EPA 8011; EPA 504	1, 2-Dibromo-3-chloropropane
GC/FID	RSK-175	Methane Ethane Ethene
GC/MS	EPA 8260B/C; EPA 524.2	1, 1, 1, 2-Tetrachloroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 1, 1-Trichloroethane
GC/MS	EPA 624; 8260B/C; EPA 524.2	1, 1, 2, 2-Tetrachloroethane
GC/MS	EPA 8260B/C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 1, 2-Trichloroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 1-Dichloroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 1-Dichloroethene
GC/MS	EPA 8260B/C; EPA 524.2	1, 1-Dichloropropene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2, 3-Trichlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2, 3-Trichloropropane
GC/MS	EPA 8260B/C	1,2,3-Trimethylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 2, 4-Trimethylbenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C; EPA 524.2	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C; EPA 524.2	1, 2-Dibromoethane (EDB)
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 2-Dichlorobenzene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 2-Dichloroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 2-Dichloropropane
GC/MS	EPA 8260B/C	1,3,5-Trichlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 3, 5-Trimethylbenzene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 3-Dichlorobenzene
GC/MS	EPA 8260B/C; EPA 524.2	1, 3-Dichloropropane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	1, 4-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 4-Dioxane
GC/MS	EPA 8260B/C	1-Chlorohexane
GC/MS	EPA 8260B/C; EPA 524.2	2, 2-Dichloropropane
GC/MS	EPA 8260B/C; EPA 524.2	2-Butanone
GC/MS	EPA 624; EPA 8260B/C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B/C; EPA 524.2	2-Chlorotoluene
GC/MS	EPA 8260B/C; EPA 524.2	2-Hexanone
GC/MS	EPA 8260B/C; EPA 524.2	4-Chlorotoluene
GC/MS	EPA 8260B/C; EPA 524.2	4-Methyl-2-pentanone
GC/MS	EPA 8260B/C; EPA 524.2	Acetone
GC/MS	EPA 8260B/C	Acetonitrile
GC/MS	EPA 624; EPA 8260B/C	Acrolein
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Acrylonitrile
GC/MS	EPA 8260B/C; EPA 524.2	Allyl chloride
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Benzene
GC/MS	EPA 8260B/C	Benzyl chloride
GC/MS	EPA 8260B/C; EPA 524.2	Bromobenzene
GC/MS	EPA 8260B/C; EPA 524.2	Bromochloromethane

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Bromodichloromethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Bromoform
GC/MS	EPA 8260B/C; EPA 524.2	Carbon disulfide
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Carbon tetrachloride
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Chlorobenzene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Chloroethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Chloroform
GC/MS	EPA 8260B/C	Chloroprene
GC/MS	EPA 8260B/C; EPA 524.2	cis-1, 2-Dichloroethene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	cis-1, 3-Dichloropropene
GC/MS	EPA 8260B/C	Cis-1,4-Dichloro-2-butene
GC/MS	EPA 8260B/C	Cyclohexane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Dibromochloromethane
GC/MS	EPA 8260B/C; EPA 524.2	Dibromomethane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Dichlorodifluoromethane
GC/MS	EPA 8260B/C; EPA 524.2	Diethyl ether
GC/MS	EPA 8260B/C	Di-isopropylether
GC/MS	EPA 8260B/C; EPA 524.2	Ethyl methacrylate
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Ethylbenzene
GC/MS	EPA 8260B/C	Ethyl-t-butylether
GC/MS	EPA 8260B/C; EPA 524.2	Hexachlorobutadiene
GC/MS	EPA 8260B/C	Iodomethane
GC/MS	EPA 8260B/C	Isobutyl alcohol
GC/MS	EPA 8260B/C	Isopropyl alcohol
GC/MS	EPA 8260B/C; EPA 524.2	Isopropyl benzene
GC/MS	EPA 8260B/C; EPA 524.2	m p-xylenes
GC/MS	EPA 8260B/C	Methyl acetate

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C; EPA 524.2	Methacrylonitrile
GC/MS	EPA 624 / 8260B,C	Methyl bromide (Bromomethane)
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Methyl chloride (Chloromethane)
GC/MS	EPA 8260B/C; EPA 524.2	Methyl methacrylate
GC/MS	EPA 8260B/C; EPA 524.2	Methyl tert-butyl ether
GC/MS	EPA 8260B/C	Methylcyclohexane
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Methylene chloride
GC/MS	EPA 8260B/C; EPA 524.2	Naphthalene
GC/MS	EPA 8260B/C; EPA 524.2	n-Butylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	n-Propylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	o-Xylene
GC/MS	EPA 8260B/C	Pentachloroethane
GC/MS	EPA 8260B/C; EPA 524.2	p-Isopropyltoluene
GC/MS	EPA 8260B/C; EPA 524.2	Propionitrile
GC/MS	EPA 8260B/C; EPA 524.2	sec-butylbenzene
GC/MS	EPA 8260B/C; EPA 524.2	Styrene
GC/MS	EPA 8260B/C	t-Amylmethylether
GC/MS	EPA 8260B/C; EPA 524.2	tert-Butyl alcohol
GC/MS	EPA 8260B/C	tert-Butylbenzene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Tetrachloroethene (Perchloroethylene)
GC/MS	EPA 8260B/C; EPA 524.2	Tetrahydrofuran
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Toluene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	trans-1, 2-Dichloroethylene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	trans-1, 3-Dichloropropylene
GC/MS	EPA 8260B/C; EPA 524.2	trans-1, 4-Dichloro-2-butuene
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Trichloroethene (Trichloroethylene)
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Trichlorofluoromethane
GC/MS	EPA 8260B/C	Vinyl acetate

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 624; EPA 8260B/C; EPA 524.2	Vinyl chloride
GC/MS	EPA 624 / 8260B,C	Xylene
GC/MS	EPA 8260B/C SIM	1,1,1,2-Tetrachloroethane
GC/MS	8260B, C SIM	1,1,1-Trichloroethane
GC/MS	EPA 8260B/C SIM	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/C SIM	1, 1, 2-Trichloroethane
GC/MS	EPA 8260B/C SIM	1,2,3-Trichloropropane
GC/MS	EPA 8260B/C SIM	1,1-Dichloroethane
GC/MS	EPA 8260B/C SIM	1,1-Dichloroethene
GC/MS	EPA 8260B/C SIM	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/C SIM	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/C SIM	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C SIM	1,2-Dibromoethane
GC/MS	EPA 8260B/C SIM	1,2-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	1,2-Dichloroethane
GC/MS	EPA 8260B/C SIM	1,2-Dichloropropane
GC/MS	EPA 8260B/C SIM	1,3-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	1,3-Dichloropropane
GC/MS	EPA 8260B/C SIM	1,4-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	2-Hexanone
GC/MS	EPA 8260B/C SIM	4-Methyl-2-pentanone
GC/MS	EPA 8260B/C SIM	Benzene
GC/MS	EPA 8260B/C SIM	Bromodichloromethane
GC/MS	EPA 8260B/C SIM	Carbon Tetrachloride
GC/MS	EPA 8260B/C SIM	Chloroform
GC/MS	EPA 8260B/C SIM	Chloromethane
GC/MS	EPA 8260B/C SIM	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/C SIM	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/C SIM	Dibromochloromethane
GC/MS	EPA 8260B/C SIM	Ethylbenzene
GC/MS	EPA 8260B/C SIM	Isopropylbenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C SIM	Hexachlorobutadiene
GC/MS	EPA 8260B/C SIM	Methylcyclohexane
GC/MS	EPA 8260B/C SIM	m,p-Xylene
GC/MS	EPA 8260B/C SIM	o-Xylene
GC/MS	EPA 8260B/C SIM	Tetrachloroethene
GC/MS	EPA 8260B/C SIM	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/C SIM	Trans-1,3-Dichloropropene
GC/MS	EPA 8260B/C SIM	Trichloroethene
GC/MS	EPA 8260B/C SIM	Trichlorofluoromethane
GC/MS	EPA 8260B/C SIM	Vinyl Chloride
GC/MS	EPA 8260B/C SIM	Xylenes (total)
GC/MS	EPA 8270C/D	1, 2, 4, 5-Tetrachlorobenzene
GC/MS	EPA 625; EPA 8270C/D	1, 2, 4-Trichlorobenzene
GC/MS	EPA 625; EPA 8270C/D	1, 2-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1, 3, 5-Trinitrobenzene
GC/MS	EPA 625; EPA 8270C/D	1, 3-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 3-Dinitrobenzene
GC/MS	EPA 625; EPA 8270C/D	1, 4-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 4-Dioxane
GC/MS	EPA 8270C/D	1, 4-Naphthoquinone
GC/MS	EPA 8270C/D	1, 4-Phenylenediamine
GC/MS	EPA 8270C/D	1-Chloronaphthalene
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	1-Naphthylamine
GC/MS	EPA 8270C/D	2, 3, 4, 6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2, 4, 5-Trochlorophenol
GC/MS	EPA 625; EPA 8270C/D	2, 4, 6-Trichlorophenol
GC/MS	EPA 625; EPA 8270C/D	2, 4-Dichlorophenol
GC/MS	EPA 625; EPA 8270C/D	2, 4-Dimethylphenol
GC/MS	EPA 625; EPA 8270C/D	2, 4-Dinitrophenol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 625; EPA 8270C/D	2, 4-Dinitrotoluene (2, 4-DNT)
GC/MS	EPA 8270C/D	2, 6-Dichlorophenol
GC/MS	EPA 625; EPA 8270C/D	2, 6-Dinitrotoluene (2, 6-DNT)
GC/MS	EPA 8270C/D	2-Acetylaminofluorene
GC/MS	EPA 625; EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 625; EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 625; EPA 8270C/D	2-Methyl-4 6-dinitrophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol
GC/MS	EPA 8270C/D	2-Naphthylamine
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 625; EPA 8270C/D	2-Nitrophenol
GC/MS	EPA 8270C/D	2-Picoline
GC/MS	EPA 8270C/D	3-Methylcholanthrene
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4-Aminobiphenyl
GC/MS	EPA 625; EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 625; EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 625; EPA 8270C/D	4-Chlorophenyl phenylether
GC/MS	EPA 8270C/D	4-Dimethyl aminoazobenzene
GC/MS	EPA 8270C/D	3, 4-Methylphenol
GC/MS	EPA 8270C/D	4-Nitroaniline
GC/MS	EPA 625; EPA 8270C/D	4-Nitrophenol
GC/MS	EPA 8270C/D	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270C/D	5-Nitro-o-toluidine
GC/MS	EPA 8270C/D	7, 12-Dimethylbenz(a)anthracene
GC/MS	EPA 8270C/D	a a-Dimethylphenethylamine
GC/MS	EPA 625; EPA 8270C/D	Acenaphthene
GC/MS	EPA 625; EPA 8270C/D	Acenaphthylene
GC/MS	EPA 8270C/D	Acetophenone

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 625; EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Aramite
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Azobenzene
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 625; EPA 8270C/D	Benzidine
GC/MS	EPA 625; EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 625; EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 625; EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 625; EPA 8270C/D	Benzo(g h i)perylene
GC/MS	EPA 625; EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	1,1-Biphenyl
GC/MS	EPA 625; EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 625; EPA 8270C/D	bis(2-Chloroethyl) ether
GC/MS	EPA 625; EPA 8270C/D	bis(2-Chloroisopropyl) ether (2, 2`-Oxybis(1-chloropropane)
GC/MS	EPA 625; EPA 8270C/D	bis(2-Ethylhexyl)adipate
GC/MS	EPA 625; EPA 8270C/D	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 625; EPA 8270C/D	Butyl benzyl phthalate
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chlorobenzilate
GC/MS	EPA 625; EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Diallate
GC/MS	EPA 8270C/D	Dibenzo(a,j)acridine
GC/MS	EPA 625; EPA 8270C/D	Dibenz(a h)anthracene
GC/MS	EPA 8270C/D	Dibenzofuran
GC/MS	EPA 8270C/D	Diethyladipate
GC/MS	EPA 625; EPA 8270C/D	Diethyl phthalate

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Dimethoate
GC/MS	EPA 625; EPA 8270C/D	Dimethyl phthalate
GC/MS	EPA 625; EPA 8270C/D	Di-n-butyl phthalate
GC/MS	EPA 625; EPA 8270C/D	Di-n-octyl phthalate
GC/MS	EPA 8270C/D	Dinoseb
GC/MS	EPA 8270C/D	Disulfoton
GC/MS	EPA 8270C/D	Ethyl methanesulfonate
GC/MS	EPA 8270C/D	Ethyl parathion
GC/MS	EPA 8270C/D	Ethyl methacrylate
GC/MS	EPA 8270C/D	Famfur
GC/MS	EPA 625; EPA 8270C/D	Fluoranthene
GC/MS	EPA 625; EPA 8270C/D	Fluorene
GC/MS	EPA 625; EPA 8270C/D	Hexachlorobenzene
GC/MS	EPA 625; EPA 8270C/D	Hexachlorobutadiene
GC/MS	EPA 625; EPA 8270C/D	Hexachlorocyclopentadiene
GC/MS	EPA 625; EPA 8270C/D	Hexachloroethane
GC/MS	EPA 8270C/D	Hexachlorophene
GC/MS	EPA 8270C/D	Hexachloropropene
GC/MS	EPA 625; EPA 8270C/D	Indeno(1, 2, 3-cd)pyrene
GC/MS	EPA 8270C/D	Isodrin
GC/MS	EPA 625; EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Isosafrole
GC/MS	EPA 8270C/D	Kepone
GC/MS	EPA 8270C/D	Methapyriline
GC/MS	EPA 8270C/D	Methy methanesulfonate
GC/MS	EPA 8270C/D	Methyl parathion
GC/MS	EPA 625; EPA 8270C/D	Naphthalene
GC/MS	EPA 625; EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	Nitroquinoline-1-oxide
GC/MS	EPA 8270C/D	n-Nitrosodiethylamine
GC/MS	EPA 625; EPA 8270C/D	n-Nitrosodimethylamine

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	n-Nitroso-di-n-butylamine
GC/MS	EPA 625; EPA 8270C/D	n-Nitrosodi-n-propylamine
GC/MS	EPA 625; EPA 8270C/D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/D	n-Nitrosomethylethylamine
GC/MS	EPA 8270C/D	n-Nitrosomorpholine
GC/MS	EPA 8270C/D	n-Nitrosopiperidine
GC/MS	EPA 8270C/D	n-Nitrosopyrrolidine
GC/MS	EPA 8270C/D	O,O,O-Triethyl phosphorothioate
GC/MS	EPA 8270C/D	o,o-Diethyl o-2pyrazinyl phosphorothioate
GC/MS	EPA 8270C/D	o-Toluidine
GC/MS	EPA 8270C/D	Pentachlorobenzene
GC/MS	EPA 8270C/D	Pentachloronitrobenzene
GC/MS	EPA 625; EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 8270C/D	Phenacetin
GC/MS	EPA 625; EPA 8270C/D	Phenanthrene
GC/MS	EPA 625; EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Phorate
GC/MS	EPA 8270C/D	Pronamide
GC/MS	EPA 625; EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/MS	EPA 8270C/D	Safrole
GC/MS	EPA 8270C/D	Sulfotepp
GC/MS	EPA 8270C/D	Thionazin
GC/MS	EPA 625; EPA 8270C/D	3, 3’-Dichlorobenzidine
GC/MS	EPA 8270C/D	3, 3’-Dimethylbenzidine
GC/MS	EPA 8270C/D SIM	1,1'-Biphenyl
GC/MS	EPA 8270C/D SIM	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D SIM	1,4-Dioxane
GC/MS	EPA 8270C/D SIM	1-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2,2'-Oxybis(1-chloropropane
GC/MS	EPA 8270C/D SIM	2,3,4,6-Tetrachlorophenol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4-Dichlorophenol
GC/MS	EPA 8270C/D SIM	2,4-Dimethylphenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrophenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2-Chloronaphthalene
GC/MS	EPA 8270C/D SIM	2-Chlorophenol
GC/MS	EPA 8270C/D SIM	2-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2-Methylphenol
GC/MS	EPA 8270C/D SIM	2-Nitroaniline
GC/MS	EPA 8270C/D SIM	2-Nitrophenol
GC/MS	EPA 8270C/D SIM	3&4-Methylphenol
GC/MS	EPA 8270C/D SIM	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C/D SIM	3-Nitroaniline
GC/MS	EPA 8270C/D SIM	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/D SIM	4-Bromophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D SIM	4-Chloroaniline
GC/MS	EPA 8270C/D SIM	4-Chlorophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Nitroaniline
GC/MS	EPA 8270C/D SIM	4-Nitrophenol
GC/MS	EPA 8270C/D SIM	Acenaphthene
GC/MS	EPA 8270C/D SIM	Acenaphthylene
GC/MS	EPA 8270C/D SIM	Acetophenone
GC/MS	EPA 8270C/D SIM	Anthracene
GC/MS	EPA 8270C/D SIM	Atrazine
GC/MS	EPA 8270C/D SIM	Benzaldehyde
GC/MS	EPA 8270C/D SIM	Benzo(a)anthracene
GC/MS	EPA 8270C/D SIM	Benzo(a)pyrene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D SIM	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D SIM	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethoxy)methane
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethyl)ether
GC/MS	EPA 8270C/D SIM	Bis(2-ethylhexyl)phthalate
GC/MS	EPA 8270C/D SIM	Butylbenzylphthalate
GC/MS	EPA 8270C/D SIM	Caprolactam
GC/MS	EPA 8270C/D SIM	Carbazole
GC/MS	EPA 8270C/D SIM	Chrysene
GC/MS	EPA 8270C/D SIM	Dibenzo(a,h)anthracene
GC/MS	EPA 8270C/D SIM	Dibenzofuran
GC/MS	EPA 8270C/D SIM	Diethylphthalate
GC/MS	EPA 8270C/D SIM	Dimethyl phthalate
GC/MS	EPA 8270C/D SIM	Di-n-butylphthalate
GC/MS	EPA 8270C/D SIM	Di-n-octylphthalate
GC/MS	EPA 8270C/D SIM	Fluoranthene
GC/MS	EPA 8270C/D SIM	Fluorene
GC/MS	EPA 8270C/D SIM	Hexachlorobenzene
GC/MS	EPA 8270C/D SIM	Hexachlorobutadiene
GC/MS	EPA 8270C/D SIM	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/D SIM	Hexachloroethane
GC/MS	EPA 8270C/D SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D SIM	Isophorone
GC/MS	EPA 8270C/D SIM	Naphthalene
GC/MS	EPA 8270C/D SIM	Nitrobenzene
GC/MS	EPA 8270C/D SIM	n-Nitroso-di-n-propylamine
GC/MS	EPA 8270C/D SIM	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/D SIM	Pentachlorophenol
GC/MS	EPA 8270C/D SIM	Phenanthrene
GC/MS	EPA 8270C/D SIM	Phenol

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	Pyrene
HPLC/UV	EPA 8330A/B	1, 3, 5-Trinitrobenzene
HPLC/UV	EPA 8330A/B	1, 3-Dinitrobenzene
HPLC/UV	EPA 8330A/B	2, 4, 6-Trinitrotoluene
HPLC/UV	EPA 8330A/B	2, 4-Dinitrotoluene
HPLC/UV	EPA 8330A/B	2, 6-Dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Amino-4, 6 -Dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Nitrotoluene
HPLC/UV	EPA 8330A/B	3-Nitrotoluene
HPLC/UV	EPA 8330A/B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/B	4-Amino-2, 6-Dinitrotoluene
HPLC/UV	EPA 8330A/B	4-Nitrotoluene
HPLC/UV	EPA 8330A/B	Ethylene glycol dinitrate (EGDN)
HPLC/UV	EPA 8330A/B	Hexahydro-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	EPA 8330A/B	Nitrobenzene
HPLC/UV	EPA 8330A MOD	Nitroglycerin
HPLC/UV	EPA 8330B	Nitroglycerin
HPLC/UV	EPA 8330A/B	Octahydro-1, 3, 5, 7-tetrazocine (HMX)
HPLC/UV	EPA 8330A/B	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	EPA 8330A/B	Tetryl
CVAA	EPA 245.1; EPA 7470A	Mercury
CVAF	EPA 1631E	Low Level Mercury
ICP/AES	EPA 200.7; EPA 6010B/C	Aluminum
ICP/AES	EPA 200.7; EPA 6010B/C	Antimony
ICP/AES	EPA 200.7; EPA 6010B/C	Arsenic
ICP/AES	EPA 200.7; EPA 6010B/C	Barium
ICP/AES	EPA 200.7; EPA 6010B/C	Beryllium
ICP/AES	EPA 200.7; EPA 6010B/C	Boron
ICP/AES	EPA 200.7; EPA 6010B/C	Cadmium
ICP/AES	EPA 200.7; EPA 6010B/C	Calcium
ICP/AES	EPA 200.7; EPA 6010B/C	Chromium

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/AES	EPA 200.7; EPA 6010B/C	Cobalt
ICP/AES	EPA 200.7; EPA 6010B/C	Copper
ICP/AES	EPA 200.7; EPA 6010B/C	Iron
ICP/AES	EPA 200.7; EPA 6010B/C	Lead
ICP/AES	EPA 200.7; EPA 6010B/C	Magnesium
ICP/AES	EPA 200.7; EPA 6010B/C	Manganese
ICP/AES	EPA 200.7; EPA 6010B/C	Molybdenum
ICP/AES	EPA 200.7; EPA 6010B/C	Nickel
ICP/AES	EPA 200.7; EPA 6010B/C	Potassium
ICP/AES	EPA 200.7; EPA 6010B/C	Selenium
ICP/AES	EPA 200.7; EPA 6010B/C	Silicon
ICP/AES	EPA 200.7; EPA 6010B/C	Silver
ICP/AES	EPA 200.7; EPA 6010B/C	Sodium
ICP/AES	EPA 6010B/C	Strontium
ICP/AES	EPA 200.7; EPA 6010B/C	Thallium
ICP/AES	EPA 200.7; EPA 6010B/C	Tin
ICP/AES	EPA 200.7; EPA 6010B/C	Titanium
ICP/AES	EPA 200.7; EPA 6010B/C	Vanadium
ICP/AES	EPA 200.7; EPA 6010B/C	Zinc
ICP/MS	EPA 200.8; EPA 6020A	Aluminum
ICP/MS	EPA 200.8; EPA 6020A	Antimony
ICP/MS	EPA 200.8; EPA 6020A	Arsenic
ICP/MS	EPA 200.8; EPA 6020A	Barium
ICP/MS	EPA 200.8; EPA 6020A	Beryllium
ICP/MS	EPA 200.8; EPA 6020A	Boron
ICP/MS	EPA 200.8; EPA 6020A	Cadmium
ICP/MS	EPA 200.8; EPA 6020A	Calcium
ICP/MS	EPA 200.8; EPA 6020A	Chromium
ICP/MS	EPA 200.8; EPA 6020A	Cobalt
ICP/MS	EPA 200.8; EPA 6020A	Copper
ICP/MS	EPA 200.8; EPA 6020A	Iron

Non-Potable Water		
Technology	**Method**	**Analyte**
ICP/MS	EPA 200.8; EPA 6020A	Lead
ICP/MS	EPA 200.8; EPA 6020A	Magnesium
ICP/MS	EPA 200.8; EPA 6020A	Manganese
ICP/MS	EPA 200.8; EPA 6020A	Molybdenum
ICP/MS	EPA 200.8; EPA 6020A	Nickel
ICP/MS	EPA 200.8; EPA 6020A	Potassium
ICP/MS	EPA 200.8; EPA 6020A	Selenium
ICP/MS	EPA 200.8; EPA 6020A	Silicon
ICP/MS	EPA 200.8; EPA 6020A	Silver
ICP/MS	EPA 200.8; EPA 6020A	Sodium
ICP/MS	EPA 6020A	Strontium
ICP/MS	EPA 200.8; EPA 6020A	Thallium
ICP/MS	EPA 200.8; EPA 6020A	Tin
ICP/MS	EPA 200.8; EPA 6020A	Titanium
ICP/MS	EPA 200.8; EPA 6020A	Tungsten
ICP/MS	EPA 200.8	Uranium
ICP/MS	EPA 200.8; EPA 6020A	Vanadium
ICP/MS	EPA 200.8; EPA 6020A	Zinc
IC	EPA 300.0; EPA 9056A	Bromide
IC	EPA 300.0; EPA 9056A	Chloride
IC	EPA 300.0; EPA 9056A	Fluoride
IC	EPA 300.0; EPA 9056A	Nitrate as N
IC	EPA 300.0; EPA 9056A	Nitrite as N
IC	EPA 300.0; EPA 9056A	Nitrate + Nitrite
IC	EPA 300.0; EPA 9056A	Orthophosphate as P
IC	EPA 300.0; EPA 9056A	Sulfate
IC	SOP CA-776	Lactic Acid
IC	SOP CA-776	Acetic Acid
IC	SOP CA-776	Propionic Acid
IC	SOP CA-776	Formic Acid
IC	SOP CA-776	Butyric Acid

Non-Potable Water		
Technology	**Method**	**Analyte**
IC	SOP CA-776	Pyruvic Acid
IC	SOP CA-776	i-Pentanoic Acid
IC	SOP CA-776	Pentanoic Acid
IC	SOP CA-776	i-Hexanoic Acid
IC	SOP CA-776	Hexanoic Acid
Titration	EPA 310.1; SM 2320B	Alkalinity
Caculation	SM 2340B	Hardness
Gravimetric	EPA 1664A; EPA 9070A	Oil and Grease, Oil and Grease with SGT
Gravimetric	SM 2540B/C/D	Solids
ISE	EPA 120.1; SM 2510B	Conductivity
ISE	SM 2520B	Practical Salinity
ISE	SM 4500F- C	Fluoride
ISE	SM 4500H+ B	pH
ISE	SM 5210B	TBOD / CBOD
Physical	EPA 1010A	Ignitability
Physical	EPA 9040C	pH
Titration	SM 2340C	Hardness
Titration	SM 4500SO_3 B	Sulfite
Titration	EPA 9034; SM 4500S^{2-} F	Sulfide
Titration	EPA SW-846 Chapter 7.3.4	Reactive Sulfide
IR	EPA 9060A; SM 5310B	Total organic carbon
Turbidimetric	EPA 180.1; SM 2130B	Turbidity
Turbidimetric	EPA 9038; ASTM 516-02	Sulfate
UV/VIS	EPA 335.4; EPA 9012B; SM 4500-CN G	Amenable cyanide
UV/VIS	EPA 350.1; SM 4500NH3 H	Ammonia as N
UV/VIS	SM 3500Fe D	Ferrous Iron
UV/VIS	EPA 351.2	Kjeldahl nitrogen - total
UV/VIS	EPA 353.2; SM 4500NO3 F	Nitrate + Nitrite
UV/VIS	EPA 353.2; SM 4500NO3 F	Nitrate as N
UV/VIS	EPA 353.2; SM 4500NO3 F	Nitrite as N
UV/VIS	EPA 365.2; SM 4500P E	Orthophosphate as P

Non-Potable Water		
Technology	**Method**	**Analyte**
UV/VIS	EPA 365.4	Phosphorus total
UV/VIS	EPA 821/R-91-100	AVS-SEM
UV/VIS	EPA 410.4	COD
UV/VIS	EPA 420.1; EPA 9065	Total Phenolics
UV/VIS	SM 4500Cl G	Total Residual Chlorine
UV/VIS	SM 5540C	MBAS
UV/VIS	EPA 7196A; SM 3500-Cr D	Chromium VI
UV/VIS	EPA 9012B; EPA 335.4	Total Cyanide
UV/VIS	EPA 9251; SM 4500Cl E	Chloride
UV/VIS	EPA SW-846 Chapter 7.3.4	Reactive Cyanide

Preparation	Method	Type
Cleanup Methods	EPA 3640A	Gel Permeation Clean-up
Cleanup Methods	EPA 3630C	Silica Gel
Cleanup Methods	EPA 3660B	Sulfur Clean-Up
Cleanup Methods	EPA 3665A	Sulfuric Acid Clean-Up
Organic Preparation	EPA 3510C	Separatory Funnel Extraction
Organic Preparation	EPA 3520C	Continuous Liquid-Liquid Extraction
Inorganic Preparation	EPA 3010A	Hotblock
Volatile Organic Preparation	EPA 5030C	Purge and Trap

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	2,4`-DDD
GC/ECD	EPA 8081B	2,4`-DDE
GC/ECD	EPA 8081B	2,4`-DDT
GC/ECD	EPA 8081B	4, 4`-DDD
GC/ECD	EPA 8081B	4, 4`-DDE
GC/ECD	EPA 8081B	4, 4`-DDT
GC/ECD	EPA 8081B	Aldrin

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081B	alpha-BHC (alpha-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	Alpha-Chlordane
GC/ECD	EPA 8081B	beta-BHC (beta-Hexachlorocyclohexane)
GC/ECD	EPA 608; EPA 8081B	Chlordane (tech.)
GC/ECD	EPA 8081B	Cis-Nonachlor
GC/ECD	EPA 8081B	delta-BHC
GC/ECD	EPA 8081B	Dieldrin
GC/ECD	EPA 8081B	Endosulfan I
GC/ECD	EPA 8081B	Endosulfan II
GC/ECD	EPA 8081B	Endosulfan sulfate
GC/ECD	EPA 8081B	Endrin
GC/ECD	EPA 8081B	Endrin aldehyde
GC/ECD	EPA 8081B	Endrin Ketone
GC/ECD	EPA 8081B	gamma-BHC (Lindane gamma-Hexachlorocyclohexane)
GC/ECD	EPA 8081B	gamma-Chlordane
GC/ECD	EPA 8081B	Heptachlor
GC/ECD	EPA 8081B	Heptachlor epoxide
GC/ECD	EPA 8081B	Hexachlorobenzene
GC/ECD	EPA 8081B	Methoxychlor
GC/ECD	EPA 8081B	Mirex
GC/ECD	EPA 8081B	Oxychlordane
GC/ECD	EPA 8081B	Toxaphene (Chlorinated camphene)
GC/ECD	EPA 8081B	Trans-Nonachlor
GC/ECD	EPA 8082A	Aroclor-1016 (PCB-1016)
GC/ECD	EPA 8082A	Aroclor-1221 (PCB-1221)
GC/ECD	EPA 8082A	Aroclor-1232 (PCB-1232)
GC/ECD	EPA 8082A	Aroclor-1242 (PCB-1242)
GC/ECD	EPA 8082A	Aroclor-1248 (PCB-1248)
GC/ECD	EPA 8082A	Aroclor-1254 (PCB-1254)
GC/ECD	EPA 8082A	Aroclor-1260 (PCB-1260)
GC/ECD	EPA 8082A MOD	Aroclor-1262 (PCB-1262)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A MOD	Aroclor-1268 (PCB-1268)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5, 5', 6-Nonachlorobiphenyl (BZ 206)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5, 6-Octachlorobiphenyl (BZ 195)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4', 5-Heptachlorobiphenyl (BZ 170)
GC/ECD	EPA 8082A	2, 2', 3, 3', 4, 4'-Hexachlorobiphenyl (BZ 128)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 180)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5', 6-Heptachlorobiphenyl (BZ 183)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 5-Hexachlorobiphenyl (BZ 138)
GC/ECD	EPA 8082A	2, 2', 3, 4, 4', 6, 6'-Heptachlorobiphenyl (BZ 184)
GC/ECD	EPA 8082A	2, 2', 3, 4', 5, 5', 6-Heptachlorobiphenyl (BZ 187)
GC/ECD	EPA 8082A	2, 2', 3, 4, 5'-Pentachlorobiphenyl (BZ 87)
GC/ECD	EPA 8082A	2, 2', 3, 5'-Tetrachlorobiphenyl (BZ 44)
GC/ECD	EPA 8082A	2, 2', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 153)
GC/ECD	EPA 8082A	2, 2', 4, 5, 5'-Pentachlorobiphenyl (BZ 101)
GC/ECD	EPA 8082A	2, 2', 4, 5-Tetrachlorobiphenyl (BZ 48)
GC/ECD	EPA 8082A	2, 2', 4, 5'-Tetrachlorobiphenyl (BZ 49)
GC/ECD	EPA 8082A	2, 2', 5, 5'-Tetrachlorobiphenyl (BZ 52)
GC/ECD	EPA 8082A	2, 2', 5-Trichlorobiphenyl (BZ 18)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5-Hexachlorobiphenyl (BZ 156)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5'-Hexachlorobiphenyl (BZ 157)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4'-Pentachlorobiphenyl (BZ 105)
GC/ECD	EPA 8082A	2, 3, 3', 4, 4', 5, 5'-Heptachlorobiphenyl (BZ 189)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 167)
GC/ECD	EPA 8082A	2, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 118)
GC/ECD	EPA 8082A	2, 3', 4, 4',5'-Pentachlorobiphenyl (BZ 123)
GC/ECD	EPA 8082A	2, 3', 4, 4'-Tetrachlorobiphenyl (BZ 66)
GC/ECD	EPA 8082A	2, 3, 4, 4', 5-Pentachlorobiphenyl (BZ 114)
GC/ECD	EPA 8082A	2, 4, 4'-Trichlorobiphenyl (BZ 28)
GC/ECD	EPA 8082A	2, 4'-Dichlorobiphenyl (BZ 8)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5, 5'-Hexachlorobiphenyl (BZ 169)
GC/ECD	EPA 8082A	3, 3', 4, 4', 5-Pentachlorobiphenyl (BZ 126)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8082A	3, 3', 4, 4'-Tetrachlorobiphenyl (BZ 77)
GC/ECD	EPA 8082A	3, 4, 4', 5-Tetrachlorobiphenyl (BZ 81)
GC/ECD	EPA 8082A	Decachlorobiphenyl (BZ 209)
GC/ECD	EPA 8151A	2, 4, 5-T
GC/ECD	EPA 8151A	2, 4-D
GC/ECD	EPA 8151A	2, 4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichloroprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP
GC/ECD	EPA 8151A	Pentachlorophenol
GC/ECD	EPA 8151A	Silvex (2, 4, 5-TP)
GC/FID	EPA 8015C/D	Diesel range organics (DRO)
GC/FID	EPA 8015C/D	Total Petroleum Hydrocarbons (TPH)
GC/FID	EPA 8015C/D	Gasoline range organics (GRO)
GC/FID/PID	MA DEP VPH	Volatile Organic Hydrocarbons
GC/FID	MA DEP EPH	Extractable Petroleum Hydrocarbons
GC/FID	MA DEP EPH EPA 3546	Extractable Petroleum Hydrocarbons Microwave Extraction Preparation
GC/FID	CT-ETPH	Total Petroleum Hydrocarbons
GC/FID	TNRCC Method 1005	Total Petroleum Hydrocarbons
GC/FID	FL-PRO	Petroleum Range Organics
GC/ECD	EPA 8011	1, 2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C	1, 1, 1, 2-Tetrachloroethane
GC/MS	EPA 8260B/C	1,1,2-Trichloro-1,2,2-trifluoroethane
GC/MS	EPA 8260B/C	1, 1, 1-Trichloroethane
GC/MS	EPA 8260B/C	1, 1, 2, 2-Tetrachloroethane
GC/MS	EPA 8260B/C	1, 1, 2-Trichloroethane
GC/MS	EPA 8260B/C	1, 1-Dichloroethane

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C	1, 1-Dichloroethylene
GC/MS	EPA 8260B/C	1, 1-Dichloropropene
GC/MS	EPA 8260B/C	1, 2, 3-Trichlorobenzene
GC/MS	EPA 8260B/C	1, 2, 3-Trichloropropane
GC/MS	EPA 8260B/C	1,2,3-Trimethylbenzene
GC/MS	EPA 8260B/C	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8260B/C	1, 2, 4-Trimethylbenzene
GC/MS	EPA 8260B/C	1, 2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C	1, 2-Dibromoethane
GC/MS	EPA 8260B/C	1, 2-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 2-Dichloroethane
GC/MS	EPA 8260B/C	1, 2-Dichloropropane
GC/MS	EPA 8260B/C	1,3,5-Trichlorobenzene
GC/MS	EPA 8260B/C	1, 3, 5-Trimethylbenzene
GC/MS	EPA 8260B/C	1, 3-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 3-Dichloropropane
GC/MS	EPA 8260B/C	1, 4-Dichlorobenzene
GC/MS	EPA 8260B/C	1, 4-Dioxane
GC/MS	EPA 8260B/C	1-Chlorohexane
GC/MS	EPA 8260B/C	2, 2-Dichloropropane
GC/MS	EPA 8260B/C	2-Butanone
GC/MS	EPA 8260B/C	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B/C	2-Chlorotoluene
GC/MS	EPA 8260B/C	2-Hexanone
GC/MS	EPA 8260B/C	4-Chlorotoluene
GC/MS	EPA 8260B/C	4-Methyl-2-pentanone
GC/MS	EPA 8260B/C	Acetone
GC/MS	EPA 8260B/C	Acetonitrile
GC/MS	EPA 8260B/C	Acrolein
GC/MS	EPA 8260B/C	Acrylonitrile
GC/MS	EPA 8260B/C	Allyl chloride

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C	Benzene
GC/MS	EPA 8260B/C	Benzyl chloride
GC/MS	EPA 8260B/C	Bromobenzene
GC/MS	EPA 8260B/C	Bromochloromethane
GC/MS	EPA 8260B/C	Bromodichloromethane
GC/MS	EPA 8260B/C	Bromoform
GC/MS	EPA 8260B/C	Carbon disulfide
GC/MS	EPA 8260B/C	Carbon tetrachloride
GC/MS	EPA 8260B/C	Chlorobenzene
GC/MS	EPA 8260B/C	Chloroethane
GC/MS	EPA 8260B/C	Chloroform
GC/MS	EPA 8260B/C	Chloroprene
GC/MS	EPA 8260B/C	cis-1, 2-Dichloroethene
GC/MS	EPA 8260B/C	cis-1, 3-Dichloropropene
GC/MS	EPA 8260B/C	cis-1,3-Dichloro-2-butene
GC/MS	EPA 8260B/C	Cyclohexane
GC/MS	EPA 8260B/C	Dibromochloromethane
GC/MS	EPA 8260B/C	Dibromomethane
GC/MS	EPA 8260B/C	Dichlorodifluoromethane
GC/MS	EPA 8260B/C	Diethyl ether
GC/MS	EPA 8260B/C	Di-isopropylether
GC/MS	EPA 8260B/C	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B/C	Ethyl methacrylate
GC/MS	EPA 8260B/C	Ethylbenzene
GC/MS	EPA 8260B/C	Ethyl-t-butylether
GC/MS	EPA 8260B/C	Hexachlorobutadiene
GC/MS	EPA 8260B/C	Iodomethane
GC/MS	EPA 8260B/C	Isobutyl alcohol
GC/MS	EPA 8260B/C	Isopropyl alcohol
GC/MS	EPA 8260B/C	Isopropyl benzene
GC/MS	EPA 8260B/C	m p-xylenes

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B, C	Methyl acetate
GC/MS	EPA 8260B/C	Methacrylonitrile
GC/MS	EPA 8260B/C	Methyl bromide (Bromomethane)
GC/MS	EPA 8260B/C	Methyl chloride (Chloromethane)
GC/MS	EPA 8260B/C	Methyl methacrylate
GC/MS	EPA 8260B/C	Methyl tert-butyl ether
GC/MS	EPA 8260B/C	Methylcyclohexane
GC/MS	EPA 8260B/C	Methylene chloride
GC/MS	EPA 8260B/C	Naphthalene
GC/MS	EPA 8260B/C	n-Butylbenzene
GC/MS	EPA 8260B/C	n-proplybenzene
GC/MS	EPA 8260B/C	o-Xylene
GC/MS	EPA 8260B/C	pentachloroethane
GC/MS	EPA 8260B/C	p-Isopropyltoluene
GC/MS	EPA 8260B/C	Propionitrile
GC/MS	EPA 8260B/C	sec-butylbenzene
GC/MS	EPA 8260B/C	Styrene
GC/MS	EPA 8260B/C	t-Amylmethylether
GC/MS	EPA 8260B/C	tert-Butyl alcohol
GC/MS	EPA 8260B/C	tert-Butylbenzene
GC/MS	EPA 8260B/C	Tetrachloroethylene (Perchloroethylene)
GC/MS	EPA 8260B/C	Tetrahydrofuran
GC/MS	EPA 8260B/C	Toluene
GC/MS	EPA 8260B/C	trans-1, 2-Dichloroethylene
GC/MS	EPA 8260B/C	trans-1, 3-Dichloropropylene
GC/MS	EPA 8260B/C	Trans-1, 4-Dichloro-2-butuene
GC/MS	EPA 8260B/C	Trichloroethene (Trichloroethylene)
GC/MS	EPA 8260B/C	Trichlorofluoromethane
GC/MS	EPA 8260B/C	Vinyl acetate
GC/MS	EPA 8260B/C	Vinyl chloride
GC/MS	EPA 8260B/C	Xylene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C SIM	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B/C SIM	1,1,1-Trichloroethane
GC/MS	EPA 8260B/C SIM	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B/C SIM	1, 1, 2-Trichloroethane
GC/MS	EPA 8260B/C SIM	1,2,3-Trichloropropane
GC/MS	EPA 8260B/C SIM	1,1-Dichloroethane
GC/MS	EPA 8260B/C SIM	1,1-Dichloroethene
GC/MS	EPA 8260B/C SIM	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B/C SIM	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B/C SIM	1,2-Dibromo-3-chloropropane
GC/MS	EPA 8260B/C SIM	1,2-Dibromoethane
GC/MS	EPA 8260B/C SIM	1,2-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	1,2-Dichloroethane
GC/MS	EPA 8260B/C SIM	1,2-Dichloropropane
GC/MS	EPA 8260B/C SIM	1,3-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	1,3-Dichloropropane
GC/MS	EPA 8260B/C SIM	1,4-Dichlorobenzene
GC/MS	EPA 8260B/C SIM	2-Hexanone
GC/MS	EPA 8260B/C SIM	4-Methyl-2-pentanone
GC/MS	EPA 8260B/C SIM	Benzene
GC/MS	EPA 8260B/C SIM	Bromodichloromethane
GC/MS	EPA 8260B/C SIM	Carbon Tetrachloride
GC/MS	EPA 8260B/C SIM	Chloroform
GC/MS	EPA 8260B/C SIM	Chloromethane
GC/MS	EPA 8260B/C SIM	cis-1,2-Dichloroethene
GC/MS	EPA 8260B/C SIM	cis-1,3-Dichloropropene
GC/MS	EPA 8260B/C SIM	Dibromochloromethane
GC/MS	EPA 8260B/C SIM	Ethylbenzene
GC/MS	EPA 8260B/C SIM	Isopropylbenzene
GC/MS	EPA 8260B/C SIM	Hexachlorobutadiene
GC/MS	EPA 8260B/C SIM	Methylcyclohexane

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B/C SIM	m,p-Xylene
GC/MS	EPA 8260B/C SIM	o-Xylene
GC/MS	EPA 8260B/C SIM	Tetrachloroethene
GC/MS	EPA 8260B/C SIM	trans-1,2-Dichloroethene
GC/MS	EPA 8260B/C SIM	Trans-1,3-Dichloropropene
GC/MS	EPA 8260B/C SIM	Trichloroethene
GC/MS	EPA 8260B/C SIM	Trichlorofluoromethane
GC/MS	EPA 8260B/C SIM	Vinyl Chloride
GC/MS	EPA 8260B/C SIM	Xylenes (total)
GC/MS	EPA 8270C/D	1, 2, 4, 5-Tetrachlorobenzene
GC/MS	EPA 8270C/D	1, 2, 4-Trichlorobenzene
GC/MS	EPA 8270C/D	1, 2-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1, 3, 5-Trinitrobenzene
GC/MS	EPA 8270C/D	1, 3-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 3-Dinitrobenzene
GC/MS	EPA 8270C/D	1, 4-Dichlorobenzene
GC/MS	EPA 8270C/D	1, 4-Dioxane
GC/MS	EPA 8270C/D	1, 4-Naphthoquinone
GC/MS	EPA 8270C/D	1, 4-Phenylenediamine
GC/MS	EPA 8270C/D	1,1-Biphenyl
GC/MS	EPA 8270C/D	1-Chloronaphthalene
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	1-Naphthylamine
GC/MS	EPA 8270C/D	2, 3, 4, 6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2, 4, 5-Trochlorophenol
GC/MS	EPA 8270C/D	2, 4, 6-Trichlorophenol
GC/MS	EPA 8270C/D	2, 4-Dichlorophenol
GC/MS	EPA 8270C/D	2, 4-Dimethylphenol
GC/MS	EPA 8270C/D	2, 4-Dinitrophenol
GC/MS	EPA 8270C/D	2, 4-Dinitrotoluene (2 4-DNT)

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	2, 6-Dichlorophenol
GC/MS	EPA 8270C/D	2, 6-Dinitrotoluene (2 6-DNT)
GC/MS	EPA 8270C/D	2-Acetylaminofluorene
GC/MS	EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 8270C/D	2-Methyl-4, 6-dinitrophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol
GC/MS	EPA 8270C/D	2-Naphthylamine
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 8270C/D	2-Nitrophenol
GC/MS	EPA 8270C/D	2-Picoline
GC/MS	EPA 8270C/D	3, 3`-Dichlorobenzidine
GC/MS	EPA 8270C/D	3, 3'-Dimethylbenzidine
GC/MS	EPA 8270C/D	3,4-Methylphenol
GC/MS	EPA 8270C/D	3-Methylcholanthrene
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4-Aminobiphenyl
GC/MS	EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 8270C/D	4-Chlorophenyl phenylether
GC/MS	EPA 8270C/D	4-Dimethyl aminoazobenzene
GC/MS	EPA 8270C/D	4-Nitroaniline
GC/MS	EPA 8270C/D	4-Nitrophenol
GC/MS	EPA 8270C/D	4-Nitroquinoline-1-oxide
GC/MS	EPA 8270C/D	5-Nitro-o-toluidine
GC/MS	EPA 8270C/D	7,12-Dimethylbenz(a)anthracene
GC/MS	EPA 8270C/D	a a-Dimethylphenethylamine
GC/MS	EPA 8270C/D	Acenaphthene
GC/MS	EPA 8270C/D	Acenaphthylene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Acetophenone
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Aramite
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Azobenzene
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 8270C/D	Benzidine
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D	Benzo(g h i)perylene
GC/MS	EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270C/D	bis(2-Chloroethyl) ether
GC/MS	EPA 8270C/D	bis(2-Chloroisopropyl) ether (2, 2`-Oxybis(1-chloropropane))
GC/MS	EPA 8270C/D	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 625; EPA 8270C/D	Bis(2-Ethylhexyl)adipate
GC/MS	EPA 8270C/D	Butyl benzyl phthalate
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chlorobenzilate
GC/MS	EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Diallate
GC/MS	EPA 8270C/D	Dibenz(a h)anthracene
GC/MS	EPA 8270C/D	Dibenzo(a,j)acridine
GC/MS	EPA 8270C/D	Dibenzofuran
GC/MS	EPA 8270C/D	Diethyl phthalate
GC/MS	EPA 8270C/D	Diethyladipate

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Dimethoate
GC/MS	EPA 8270C/D	Dimethyl phthalate
GC/MS	EPA 8270C/D	Di-n-butyl phthalate
GC/MS	EPA 8270C/D	Di-n-octyl phthalate
GC/MS	EPA 8270C/D	Dinoseb
GC/MS	EPA 8270C/D	Disulfoton
GC/MS	EPA 8270C/D	Ethyl methacrylate
GC/MS	EPA 8270C/D	Ethyl methanesulfonate
GC/MS	EPA 8270C/D	Ethyl parathion
GC/MS	EPA 8270C/D	Famfur
GC/MS	EPA 8270C/D	Fluoranthene
GC/MS	EPA 8270C/D	Fluorene
GC/MS	EPA 8270C/D	Hexachlorobenzene
GC/MS	EPA 8270C/D	Hexachlorobutadiene
GC/MS	EPA 8270C/D	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/D	Hexachloroethane
GC/MS	EPA 8270C/D	Hexachlorophene
GC/MS	EPA 8270C/D	Hexachloropropene
GC/MS	EPA 8270C/D	Indeno(1, 2, 3-cd)pyrene
GC/MS	EPA 8270C/D	Isodrin
GC/MS	EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Isosafrole
GC/MS	EPA 8270C/D	Kepone
GC/MS	EPA 8270C/D	Methapyriline
GC/MS	EPA 8270C/D	Methyl methanesulfonate
GC/MS	EPA 8270C/D	Methyl parathion
GC/MS	EPA 8270C/D	Naphthalene
GC/MS	EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	n-Nitrosodiethylamine
GC/MS	EPA 8270C/D	n-Nitrosodimethylamine
GC/MS	EPA 8270C/D	n-Nitroso-di-n-butylamine

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270C/D	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/D	n-Nitrosomethylethylamine
GC/MS	EPA 8270C/D	n-Nitrosomorpholine
GC/MS	EPA 8270C/D	n-Nitrosopiperidine
GC/MS	EPA 8270C/D	n-Nitrosopyrrolidine
GC/MS	EPA 8270C/D	O, O, O-Triethyl phosphorothioate
GC/MS	EPA 8270C/D	o,o-Diethyl o-2-pyrazinyl phosphorothioate
GC/MS	EPA 8270C/D	o-Toluidine
GC/MS	EPA 8270C/D	Pentachlorobenzene
GC/MS	EPA 8270C/D	Pentachloronitrobenzene
GC/MS	EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 8270C/D	Phenacetin
GC/MS	EPA 8270C/D	Phenanthrene
GC/MS	EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Phorate
GC/MS	EPA 8270C/D	Pronamide
GC/MS	EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/MS	EPA 8270C/D	Safrole
GC/MS	EPA 8270C/D	Sulfotepp
GC/MS	EPA 8270C/D	Thionazin
GC/MS	EPA 8270C/D SIM	1,1'-Biphenyl
GC/MS	EPA 8270C/D SIM	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D SIM	1,4-Dioxane
GC/MS	EPA 8270C/D SIM	1-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2,2'-Oxybis(1-chloropropane
GC/MS	EPA 8270C/D SIM	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D SIM	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4,6-Trichlorophenol
GC/MS	EPA 8270C/D SIM	2,4-Dichlorophenol

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	2,4-Dimethylphenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrophenol
GC/MS	EPA 8270C/D SIM	2,4-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D SIM	2-Chloronaphthalene
GC/MS	EPA 8270C/D SIM	2-Chlorophenol
GC/MS	EPA 8270C/D SIM	2-Methylnaphthalene
GC/MS	EPA 8270C/D SIM	2-Methylphenol
GC/MS	EPA 8270C/D SIM	2-Nitroaniline
GC/MS	EPA 8270C/D SIM	2-Nitrophenol
GC/MS	EPA 8270C/D SIM	3&4-Methylphenol
GC/MS	EPA 8270C/D SIM	3,3'-Dichlorobenzidine
GC/MS	EPA 8270C/D SIM	3-Nitroaniline
GC/MS	EPA 8270C/D SIM	4,6-Dinitro-2-methylphenol
GC/MS	EPA 8270C/D SIM	4-Bromophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D SIM	4-Chloroaniline
GC/MS	EPA 8270C/D SIM	4-Chlorophenyl-phenylether
GC/MS	EPA 8270C/D SIM	4-Nitroaniline
GC/MS	EPA 8270C/D SIM	4-Nitrophenol
GC/MS	EPA 8270C/D SIM	Acenaphthene
GC/MS	EPA 8270C/D SIM	Acenaphthylene
GC/MS	EPA 8270C/D SIM	Acetophenone
GC/MS	EPA 8270C/D SIM	Anthracene
GC/MS	EPA 8270C/D SIM	Atrazine
GC/MS	EPA 8270C/D SIM	Benzaldehyde
GC/MS	EPA 8270C/D SIM	Benzo(a)anthracene
GC/MS	EPA 8270C/D SIM	Benzo(a)pyrene
GC/MS	EPA 8270C/D SIM	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D SIM	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D SIM	Benzo(k)fluoranthene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethoxy)methane
GC/MS	EPA 8270C/D SIM	Bis(2-chloroethyl)ether
GC/MS	EPA 8270C/D SIM	Bis(2-ethylhexyl)phthalate
GC/MS	EPA 8270C/D SIM	Butylbenzylphthalate
GC/MS	EPA 8270C/D SIM	Caprolactam
GC/MS	EPA 8270C/D SIM	Carbazole
GC/MS	EPA 8270C/D SIM	Chrysene
GC/MS	EPA 8270C/D SIM	Dibenzo(a,h)anthracene
GC/MS	EPA 8270C/D SIM	Dibenzofuran
GC/MS	EPA 8270C/D SIM	Diethylphthalate
GC/MS	EPA 8270C/D SIM	Dimethyl phthalate
GC/MS	EPA 8270C/D SIM	Di-n-butylphthalate
GC/MS	EPA 8270C/D SIM	Di-n-octylphthalate
GC/MS	EPA 8270C/D SIM	Fluoranthene
GC/MS	EPA 8270C/D SIM	Fluorene
GC/MS	EPA 8270C/D SIM	Hexachlorobenzene
GC/MS	EPA 8270C/D SIM	Hexachlorobutadiene
GC/MS	EPA 8270C/D SIM	Hexachlorocyclopentadiene
GC/MS	EPA 8270C/D SIM	Hexachloroethane
GC/MS	EPA 8270C/D SIM	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D SIM	Isophorone
GC/MS	EPA 8270C/D SIM	Naphthalene
GC/MS	EPA 8270C/D SIM	Nitrobenzene
GC/MS	EPA 8270C/D SIM	n-Nitroso-di-n-propylamine
GC/MS	EPA 8270C/D SIM	n-Nitrosodiphenylamine
GC/MS	EPA 8270C/D SIM	Pentachlorophenol
GC/MS	EPA 8270C/D SIM	Phenanthrene
GC/MS	EPA 8270C/D SIM	Phenol
GC/MS	EPA 8270C/D SIM	Pyrene
HPLC/UV	EPA 8330A	1 ,3, 5-Trinitrobenzene
HPLC/UV	EPA 8330A	1, 3-Dinitrobenzene

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
HPLC/UV	EPA 8330A	2, 4, 6-Trinitrotoluene
HPLC/UV	EPA 8330A	2, 4-Dinitrotoluene
HPLC/UV	EPA 8330A	2, 6-Dinitrotoluene
HPLC/UV	EPA 8330A	2-Amino-4, 6-dinitrotoluene
HPLC/UV	EPA 8330A	2-Nitrotoluene
HPLC/UV	EPA 8330A	3-Nitrotoluene
HPLC/UV	EPA 8330A	3,5-Dinitroaniline
HPLC/UV	EPA 8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A	4-Nitrotoluene
HPLC/UV	EPA 8330A	Ethylene glycol dinitrate (EGDN)
HPLC/UV	EPA 8330A	Hexahydr-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	EPA 8330A	Nitrobenzene
HPLC/UV	EPA 8330A MOD	Nitroglycerin
HPLC/UV	EPA 8330A	Octahydro-1, 3, 5, 7-tetrazocine (HMX)
HPLC/UV	EPA 8330A	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	EPA 8330A	Tetryl
HPLC/UV	8330B (W/O Soil Grinding)	1, 3, 5-Trinitrobenzene
HPLC/UV	8330B (W/O Soil Grinding)	1, 3-Dinitrobenzene
HPLC/UV	8330B (W/O Soil Grinding)	2, 4, 6-Trinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	2, 4-Dinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	2, 6-Dinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	2-Amino-4, 6 –Dinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	2-Nitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	3-Nitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	3,5-Dinitroaniline
HPLC/UV	8330B (W/O Soil Grinding)	4-Amino-2,3-Dinitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	4-Nitrotoluene
HPLC/UV	8330B (W/O Soil Grinding)	Ethylene glycol dinitrate (EGDN)
HPLC/UV	8330B (W/O Soil Grinding)	Hexahydr-1, 3, 5-trinitro-1, 3, 5-triazine (RDX)
HPLC/UV	8330B (W/O Soil Grinding)	Nitrobenzene
HPLC/UV	8330B (W/O Soil Grinding)	Nitroglycerin

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
HPLC/UV	8330B (W/O Soil Grinding)	Octahydro-1, 3, 5, 7-tetrazocine (HMX)
HPLC/UV	8330B (W/O Soil Grinding)	Pentaerythritol Tetranitrate (PETN)
HPLC/UV	8330B (W/O Soil Grinding)	Tetryl
CVAA	EPA 7471B	Mercury
CVAF	EPA 1631E	Low Level Mercury
ICP/AES	EPA 6010B/C	Aluminum
ICP/AES	EPA 6010B/C	Antimony
ICP/AES	EPA 6010B/C	Arsenic
ICP/AES	EPA 6010B/C	Barium
ICP/AES	EPA 6010B/C	Beryllium
ICP/AES	EPA 6010B/C	Boron
ICP/AES	EPA 6010B/C	Cadmium
ICP/AES	EPA 6010B/C	Calcium
ICP/AES	EPA 6010B/C	Chromium
ICP/AES	EPA 6010B/C	Cobalt
ICP/AES	EPA 6010B/C	Copper
ICP/AES	EPA 6010B/C	Iron
ICP/AES	EPA 6010B/C	Lead
ICP/AES	EPA 6010B/C	Magnesium
ICP/AES	EPA 6010B/C	Manganese
ICP/AES	EPA 6010B/C	Molybdenum
ICP/AES	EPA 6010B/C	Nickel
ICP/AES	EPA 6010B/C	Potassium
ICP/AES	EPA 6010B/C	Selenium
ICP/AES	EPA 6010B/C	Silicon
ICP/AES	EPA 6010B/C	Silver
ICP/AES	EPA 6010B/C	Sodium
ICP/AES	EPA 6010B/C	Strontium
ICP/AES	EPA 6010B/C	Thallium
ICP/AES	EPA 6010B/C	Tin
ICP/AES	EPA 6010B/C	Titanium

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
ICP/AES	EPA 6010B/C	Vanadium
ICP/AES	EPA 6010B/C	Zinc
ICP/MS	EPA 6020A	Aluminum
ICP/MS	EPA 6020A	Antimony
ICP/MS	EPA 6020A	Arsenic
ICP/MS	EPA 6020A	Barium
ICP/MS	EPA 6020A	Beryllium
ICP/MS	EPA 6020A	Boron
ICP/MS	EPA 6020A	Cadmium
ICP/MS	EPA 6020A	Calcium
ICP/MS	EPA 6020A	Chromium
ICP/MS	EPA 6020A	Cobalt
ICP/MS	EPA 6020A	Copper
ICP/MS	EPA 6020A	Iron
ICP/MS	EPA 6020A	Lead
ICP/MS	EPA 6020A	Magnesium
ICP/MS	EPA 6020A	Manganese
ICP/MS	EPA 6020A	Molybdenum
ICP/MS	EPA 6020A	Nickel
ICP/MS	EPA 6020A	Potassium
ICP/MS	EPA 6020A	Selenium
ICP/MS	EPA 6020A	Silver
ICP/MS	EPA 6020A	Sodium
ICP/MS	EPA 6020A	Strontium
ICP/MS	EPA 6020A	Thallium
ICP/MS	EPA 6020A	Tin
ICP/MS	EPA 6020A	Titanium
ICP/MS	EPA 6020A	Tungsten
ICP/MS	EPA 6020A	Vanadium
ICP/MS	EPA 6020A	Zinc
IC	EPA 9056A	Chloride

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate as N
IC	EPA 9056A	Nitrite as N
IC	EPA 9056A	Orthophosphate
IC	EPA 9056A	Sulfate
Gravimetric	EPA 9071A; EPA 9071B	Oil and Grease, Oil and Grease with SGT
Physical	EPA 1010A	Ignitability
Physical	EPA 9045D	pH
Titration	EPA SW-846 Chapter 7.3.4	Reactive Sulfide
Titration	Walkley-Black	Total Organic Carbon
IR	Lloyd Kahn	Total organic carbon
Turbidimetric	EPA 9038; ASTM 516-02	Sulfate
UV/VIS	EPA 350.1; SM 4500NH3 H	Ammonia as N
UV/VIS	EPA 9251; SM 4500Cl E	Chloride
UV/VIS	EPA SW-846 Chapter 7.3.4	Reactive Cyanide
UV/VIS	EPA 821/R-91-100	AVS-SEM
UV/VIS	SM 3500Fe D	Ferrous Iron
Cleanup Methods	EPA 3630C	Silica Gel
UV/VIS	EPA 7196	Chromium VI
UV/VIS	EPA 7196A	Chromium VI
UV/VIS	EPA 9012B	Total cyanide
Preparation	**Method**	**Type**
Preparation	EPA 1311	Toxicity Characteristic Leaching Procedure
Preparation	EPA 1312	Synthetic Precipitation Leaching Procedure
Cleanup Methods	EPA 3660B	Sulfur Clean-up
Cleanup Methods	EPA 3620C	Florsil Clean-up
Cleanup Methods	EPA 3630C	Silica Gel Clean-up
Cleanup Methods	EPA 3640A	GPC Clean-up
Organic Preparation	EPA 3540C	Soxhlet Extraction
Organic Preparation	EPA 3545A	Pressurized Fluid Extraction

Solid and Chemical Waste		
Technology	**Method**	**Analyte**
Organic Preparation	EPA 3546	Microwave Extraction Preparation for EPA 8082A, 8081B and 8270C, D
Organic Preparation	EPA 3550C	Sonication
Inorganics Preparation	EPA 3050B	Hotblock
Inorganics Preparation	EPA 3060A	Alkaline Digestion
Volatile Organics Preparation	EPA 5035/5035A	Closed System Purge and Trap

Air		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	Propene
GC/MS	EPA TO-15	1, 1, 1-Trichloroethane
GC/MS	EPA TO-15	1, 1, 2, 2-Tetrachloroethane
GC/MS	EPA TO-15	1, 1, 2-Trichloroethane
GC/MS	EPA TO-15	1, 1-Dichloroethane
GC/MS	EPA TO-15	1, 1-Dichloroethylene
GC/MS	EPA TO-15	1, 2, 4-Trichlorobenzene
GC/MS	EPA TO-15	1, 2, 4-Trimethylbenzene
GC/MS	EPA TO-15	1, 2-Dibromoethane (EDB)
GC/MS	EPA TO-15	1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon 114)
GC/MS	EPA TO-15	1, 2-Dichlorobenzene
GC/MS	EPA TO-15	1, 2-Dichloroethane
GC/MS	EPA TO-15	1, 2-Dichloroethenes (Total)
GC/MS	EPA TO-15	1, 2-Dichloropropane
GC/MS	EPA TO-15	1, 3, 5-Trimethylbenzene
GC/MS	EPA TO-15	1, 3-Butadiene
GC/MS	EPA TO-15	1, 3-Dichlorobenzene
GC/MS	EPA TO-15	1, 4-Dichlorobenzene
GC/MS	EPA TO-15	1,4-Difluorobenzene
GC/MS	EPA TO-15	1, 4-Dioxane
GC/MS	EPA TO-15	2-Butanone
GC/MS	EPA TO-15	2-Hexanone

Air		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	2-Propanol
GC/MS	EPA TO-15	4-Ethyltoluene
GC/MS	EPA TO-15	4-Methyl-2-pentanone
GC/MS	EPA TO-15	Acetone
GC/MS	EPA TO-15	Acrolein
GC/MS	EPA TO-15	Benzene
GC/MS	EPA TO-15	Benzyl chloride
GC/MS	EPA TO-15	Bromochloromethane
GC/MS	EPA TO-15	Bromodichloromethane
GC/MS	EPA TO-15	Bromoform
GC/MS	EPA TO-15	Carbon disulfide
GC/MS	EPA TO-15	Carbon tetrachloride
GC/MS	EPA TO-15	Chlorobenzene
GC/MS	EPA TO-15	Chloroethane
GC/MS	EPA TO-15	Chloroform
GC/MS	EPA TO-15	Cis-1, 2-Dichloroethene
GC/MS	EPA TO-15	Cis-1, 3-Dichloropropene
GC/MS	EPA TO-15	Cyclohexane
GC/MS	EPA TO-15	Dibromochloromethane
GC/MS	EPA TO-15	Dichlorodifluoromethane (Freon 12)
GC/MS	EPA TO-15	Ethanol
GC/MS	EPA TO-15	Ethyl acetate
GC/MS	EPA TO-15	Ethylbenzene
GC/MS	EPA TO-15	Hexachlorobutadiene
GC/MS	EPA TO-15	Isopropyl alcohol
GC/MS	EPA TO-15	m, p-Xylene
GC/MS	EPA TO-15	Methyl bromide (Bromomethane)
GC/MS	EPA TO-15	Methyl chloride (Chloromethane)
GC/MS	EPA TO-15	Methyl methacrylate
GC/MS	EPA TO-15	Methyl tert-butyl ether
GC/MS	EPA TO-15	Methylene chloride

Air		
Technology	**Method**	**Analyte**
GC/MS	EPA TO-15	Naphthalene
GC/MS	EPA TO-15	n-Heptane
GC/MS	EPA TO-15	n-Hexane
GC/MS	EPA TO-15	o-Xylene
GC/MS	EPA TO-15	Styrene
GC/MS	EPA TO-15	Tetrachloroethylene (Perchloroethylene)
GC/MS	EPA TO-15	Tetrahydrofuran
GC/MS	EPA TO-15	Toluene
GC/MS	EPA TO-15	trans-1, 2-Dichloroethylene
GC/MS	EPA TO-15	trans-1, 3-Dichloropropylene
GC/MS	EPA TO-15	Trichloroethene (Trichloroethylene)
GC/MS	EPA TO-15	Trichlorofluoromethane (Freon 11)
GC/MS	EPA TO-15	1,1,2-Trichloro1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA TO-15	Vinyl acetate
GC/MS	EPA TO-15	Vinyl chloride
GC/MS	EPA TO-15	Xylenes (Total)

Notes:

1) This laboratory offers commercial testing service.

Approved by: _____ Date: February 1, 2013

R. Douglas Leonard
Chief Technical Officer

Re-issued: 2/1/13

State of Rhode Island and Providence Plantations
DEPARTMENT OF HEALTH
Certifies

LAO00303

KATAHDIN ANALYTICAL SERVICES INC
600 TECHNOLOGY WAY
SCARBOROUGH ME 04074
Laboratory Director: DEBORAH NADEAU

for the analysis of: **Potable Water Organic Chemistry - Potable Water Inorganic Chemistry - Non-potable Water Organic Chemistry - Non-potable Water Inorganic Chemistry -**

This certificate is issued, pursuant to Rhode Island General Laws 23-16.2 and supersedes all previous Rhode Island certificates issued to this laboratory. Certification is no guarantee of the validity of the laboratory results.

This certificate is valid only when accompanied by the certificate and list of analytes and methods for which certification has been granted based upon the following out of state certification(s):

Certifying Authority	*Certification Number*	*Expiration Date*
MAINE	*ME0019*	*06/01/2014*
NH	*200112*	*04/02/2013*

KATAHDIN ANALYTICAL SERVICES INC is responsible for maintaining each of the certifications listed above. Failure to notify the Laboratory Certification Officer of any change in the status of these certifications may result in the suspension or revocation of certification. Contact the Laboratory Certification Officer to verify the current certification status of this laboratory.

Michael Fine, MD
Director of Health

Expires: 12/30/2013

THIS CERTIFICATE IS TO BE CONSPICUOUSLY DISPLAYED AT THE LABORATORY IN A LOCATION VISABLE TO THE PUBLIC

TITLE: CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035

Prepared By: GC/MS Group Date: 7/98

Approved By:

Group Supervisor: [signature] Date: 011201

Operations Manager: [signature] Date: 1/15/01

QA Officer: [signature] Date: 1.23.01

General Manager: [signature] Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 5035	Format changes, added pollution prevention, minor changes throughout	DN	1/23/01	1/30/01
02 5035	Reorganiced sections 4, 5, 6, 7 and 8.	HRC	07.02.09	07.02.09
03 5035	Editted section 6.4.3 to include the addition of 5mL of H2O to Sample	LAD	020305	020305
04 5035	Balance weighs to 0.1g grammatical corrections formatting corrections	LAD	04/06	04/06
05	Added 3585 Reference. Sections 6.1.2.3, 6.4.3 and 7.2.2: changed 20mL to 5mL.	LAD	09/08	09/09

TITLE: CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Small changes to sections 3.2, 4.22.1, and 7.1.1 to address the differences between Purge and Trap Autosamplers. Added references.	LAD	03/12	03/12

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-214-06**, titled **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-214-06**, titled **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035.**

Recipient: _____ Date: _____

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

1.0 SCOPE AND APPLICATION

This method describes a closed-system purge-and-trap process for the analysis of volatile organic compounds (VOCs) in solid materials (e.g., soils, sediments, and solid waste). While the method is designed for use on samples containing low levels of VOCs, procedures are also provided for collecting and preparing solid samples containing high concentrations of VOCs and for oily wastes. For these high concentration and oily materials, sample collection and preparation are performed using the procedures described here, and sample introduction is performed using the aqueous purge-and-trap procedure in Method 5030. These procedures may be used in conjunction with any appropriate determinative gas chromatographic procedure, including, but not limited to, Methods 8015, 8021, and 8260.

The low soil method utilizes a hermetically sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are negligible. The applicable concentration range of the low soil method is dependent on the determinative method, matrix, and compound. However, it will generally fall in the 5.0 to 200 µg/kg range.

Procedures are included for preparing high concentration samples for purging by Method 5030. High concentration samples are those containing VOC levels of >200 µg/kg.

Procedures are also included for addressing oily wastes that are soluble in a water-miscible solvent by method 3585. These samples are also purged using Method 5030.

Method 5035 can be used for most volatile organic compounds that have boiling points below 200°C and that are insoluble or slightly soluble in water. Volatile, water-soluble compounds can be included in this analytical technique. However, quantitation limits (by GC or GC/MS) are approximately ten times higher because of poor purging efficiency.

The closed-system purge-and-trap equipment employed for low concentration samples is not appropriate for soil samples preserved in the field with methanol. Such samples should be analyzed using Method 5030 (see the note in Sec. 6.2.2).

1.1 Definitions

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analyses using Methods 5030, 5035 and 3585. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Demonstration of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of soils by method 5035 to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the department manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their department manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

After analysis, partially-filled VOA vials and sample jars are returned to the appropriate refrigerators to be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

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Sample aliquots used for analysis are disposed of in accordance with SOP SD-903 and the Katahdin Hazardous Waste Management Plan and Safety Manual. The soil samples must be decanted and the soil fraction disposed of separately in compliance with Katahdin's disposal policies.

2.0 SUMMARY OF METHOD

2.1 Low concentration soil method - generally applicable to and soils and other solid samples with VOC concentrations in the range of 5.0 to 200 µg/kg. Volatile organic compounds (VOCs) are determined by collecting an approximately 5-g sample, weighed in the field at the time of collection, and placing it in a pre-weighed vial with a septum-sealed screw-cap (see Sec. 4) that already contains a stirring bar and a sodium bisulfate or organic-free laboratory reagent grade water preservative solution. If the samples are sent to the laboratory in an Encore sampling device, the laboratory extrudes the sample into this vial containing a stirring bar and a sodium bisulfate or organic-free laboratory reagent grade water preservative solution. The entire vial is then placed, unopened, into the instrument carousel. Immediately before analysis, organic-free laboratory reagent grade water, surrogates, and internal standards (if applicable) are automatically added without opening the sample vial. The vial containing the sample is heated to 40° and the volatiles purged into an appropriate trap using an inert gas combined with agitation of the sample. Purged components travel via a transfer line to a trap. When purging is complete, the trap is heated and backflushed with helium to desorb the trapped sample components into a gas chromatograph for analysis by an appropriate determinative method.

2.2 High concentration soil method - generally applicable to soils and other solid samples with VOC concentrations greater than 200 µg/kg. The sample introduction technique in Sec. 2.1 is not applicable to all samples, particularly those containing high concentrations (generally greater than 200 µg/kg) of VOCs which may overload either the volatile trapping material or exceed the working range of the determinative instrument system (e.g., GC/MS, GC/FID, GC/EC, etc.). In such instances, this method describes two sample collection options and the corresponding sample purging procedures.

2.2.1 The first option is to collect a bulk sample in a vial or other suitable container without the use of the preservative solution described in Sec. 2.1. A portion of that sample is removed from the container in the laboratory and is dispersed in a water-miscible solvent (e.g., methanol) to dissolve the volatile organic constituents. An aliquot of the solution is added to 20 mL of laboratory reagent grade water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method.

Because the procedure involves opening the vial and removing a portion of the soil, some volatile constituents may be lost during handling.

2.2.2 The second option is to collect an approximately 5-g sample in a pre-weighed vial with a septum-sealed screw-cap (see Sec 4) that contains a known aliquot of a water-miscible organic solvent (e.g., methanol). An aliquot of the solution is added to 20 mL of laboratory reagent grade water in a purge tube. Surrogates and internal standards (if applicable) are added to the solution, then purged using Method 5030, and analyzed by an appropriate determinative method.

2.3 High concentration oily waste method - generally applicable to oily samples with VOC concentrations greater than 200 µg/kg that can be diluted in a water-miscible solvent. Samples that are comprised of oils or samples that contain significant amounts of oil present additional analytical challenges. This procedure is generally appropriate for such samples when they are soluble in a water-miscible solvent.

2.3.1 After demonstrating that a test aliquot of the sample is soluble in methanol, a separate aliquot of the sample is diluted in the appropriate solvent. An aliquot of the solution is added to 20 mL of laboratory reagent grade water in a purge tube, taking care to ensure that a floating layer of oil is not present in the purge tube. Internal standards (if applicable) and surrogates are added to the solution that is then purged using Method 5030 and analyzed by an appropriate determinative method.

2.3.2 Samples that contain oily materials that are not soluble in water-miscible solvents must be prepared in n-hexandecane according to Method 3585.

3.0 INTERFERENCES

3.1 Impurities in the purge gas and from organic compounds out-gassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running method blanks. The use of non-polytetrafluoroethylene (non-PTFE) plastic coating, non-PTFE thread sealants, or flow controllers with rubber components in the purging device must be avoided, since such materials out-gas organic compounds which will be concentrated in the trap during the purge operation. These compounds will result in interferences or false positives in the determinative step.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) through the septum seal of the sample vial during shipment and storage. A trip blank prepared from organic-free laboratory

reagent grade water or water miscible solvent and carried through sampling and handling protocols serves as a check on such contamination.

3.3 Contamination by carryover can occur whenever high-concentration and low-concentration samples are analyzed in sequence. Where practical, samples with unusually high concentrations of analytes should be followed by an analysis of organic-free laboratory reagent grade water to check for cross-contamination. If the target compounds present in an unusually concentrated sample are also found to be present in the subsequent samples, the analyst must demonstrate that the compounds are not due to carryover. Conversely, if those target compounds are not present in the subsequent sample, then the analysis of organic-free laboratory reagent grade water is not necessary.

3.4 The laboratory where volatile analysis is performed should be free of solvents. The analytical and sample storage area should be isolated from all atmospheric sources of methylene chloride, otherwise random background levels will result. Since methylene chloride will permeate through PTFE tubing, all GC carrier gas lines and purge gas plumbing should be constructed of stainless steel or copper tubing. Laboratory workers' clothing previously exposed to methylene chloride fumes during common liquid/liquid extraction procedures can contribute to sample contamination. The presence of other organic solvents in the laboratory where volatile organics are analyzed will also lead to random background levels and the same precautions must be taken.

4.0 APPARATUS AND MATERIALS

4.1 Sample Containers

A standard 40 ml VOA vial is used (e.g. ESS pre-cleaned certified 40 ml clear Type I borosilicate glass vials, open-top/polypropylene with 0.125 inch septa).

4.2 Purge-and-Trap System

The purge-and-trap system consists of a unit that automatically adds water, surrogates, and internal standards to a vial containing the sample, purges the VOCs using an inert gas stream while agitating the contents of the vial, and also traps the released VOCs for subsequent desorption into the gas chromatograph. The Purge and Trap autodampler systems at Katahdin meet the following criteria:

4.2.1 The purging device should be capable of accepting a vial sufficiently large to contain a 5-g soil sample plus a magnetic stirring bar and 10 mL of water. The device must be capable of heating a soil vial to 40°C and holding it at that temperature while the inert purge gas is allowed to pass through the

sample. The device should also be capable of introducing at least 20 mL of organic-free laboratory reagent grade water into the sample vial while trapping the displaced headspace vapors. It must also be capable of agitating the sealed sample during purging, (e.g., using a magnetic stirring bar added to the vial prior to sample collection, sonication, or other means). The analytes being purged must be quantitatively transferred to an absorber trap. The trap must be capable of transferring the absorbed VOCs to the gas chromatograph (see 4.2.2).

4.2.2 A variety of traps and trapping materials may be employed with this method. The choice of trapping material may depend on the analytes of interest. Whichever trap is employed; it must demonstrate sufficient adsorption and desorption characteristics to meet the quantitation limits of all the target analytes for a given project and the QC requirements in Method 8000 and the determinative method. The most difficult analytes are generally the gases, especially dichlorodifluoromethane. The trap must be capable of desorbing the late eluting target analytes.

NOTE: Check the responses of the brominated compounds when using alternative charcoal traps (especially Vocarb 4000), as some degradation has been noted when higher desorption temperatures (especially above 240°C - 250°C) are employed. 2-Chloroethyl vinyl ether is degraded on Vocarb 4000 but performs adequately when Vocarb 3000 is used. The primary criterion, as stated above, is that all target analytes meet the sensitivity requirements for a given project.

4.2.2.1 The standard trap used in other EPA purge-and-trap methods is also acceptable. That trap is 29 - 30 cm long and has an inside diameter of at least 0.105 in. Starting from the inlet, the trap contains the equal amounts of the adsorbents listed below. It is recommended that 1.0 cm of methyl silicone-coated packing (35/60 mesh, Davison, grade 15 or equivalent) be inserted at the inlet to extend the life of the trap. If the analysis of dichlorodifluoromethane or other fluorocarbons of similar volatility is not required, then the charcoal can be eliminated and the polymer increased to fill 2/3 of the trap. If only compounds boiling above 35° are to be analyzed, both the silica gel and charcoal can be eliminated and the polymer increased to fill the entire trap.

4.2.2.1.1 2,6-Diphenylene oxide polymer - 60/80 mesh, chromatographic grade (Tenax GC or equivalent).

4.2.2.1.2 Methyl silicone packing - OV-1 (3%) on Chromosorb-W, 60/80 mesh or equivalent.

4.2.2.1.3 Coconut charcoal - Prepare from Barnebey Cheney, CA-580-26, or equivalent, by crushing through 26 mesh screen.

4.2.2.2 Trapping materials other than those listed above also may be employed, provided that they meet the specifications in Sec. 4.2.3, below.

4.2.3 The desorber for the trap must be capable of rapidly heating the trap to the temperature recommended by the trap material manufacturer, prior to the beginning of the flow of desorption gas. Several commercial desorbers (purge-and-trap units) are available.

4.3 Syringe and Syringe Valves

4.3.1 25-mL glass hypodermic syringes with Luer-Lok (or equivalent) tip (other sizes are acceptable depending on sample volume used).

4.3.2 25-µL micro syringe with a 2 inch x 0.006 inch ID, 22° bevel needle (Hamilton #702N or equivalent).

4.3.3 Micro syringes - 10-, 100-µL.

4.3.4 Syringes - 0.5-, 1.0-, and 5-mL, gas-tight.

4.4 Miscellaneous

4.4.1 Glass vials

4.4.1.1 60-mL, septum-sealed, to collect samples for screening, dry weight determination.

4.4.1.2 40-mL, screw-cap, PTFE lined, septum-sealed. Examine each vial prior to use to ensure that the vial has a flat, uniform sealing surface.

4.4.2 Top-loading balance - Capable of accurately weighing to 0.1 g.

4.4.3 Glass scintillation vials - 20-mL, with screw-caps and PTFE liners, or glass culture tubes with screw-caps and PTFE liners, for dilution of oily waste samples.

4.4.4 Volumetric flasks - Class A, 10-mL and 100-mL, with ground glass stoppers.

4.4.5 2-mL glass vials, for GC autosampler - Used for oily waste samples extracted with methanol or PEG.

4.4.6 Spatula, stainless steel - narrow enough to fit into a sample vial.

4.4.7 Disposable Pasteur pipettes.

4.4.8 Magnetic stirring bars - PTFE- or glass-coated, of the appropriate size to fit the sample vials. Consult manufacturer's recommendation for specific stirring bars. Stirring bars may be reused, provided that they are thoroughly cleaned between uses. Consult the manufacturers of the purging device and the stirring bars for suggested cleaning procedures.

4.5 Field Sampling Equipment

4.5.1 EnCore™ sampler - (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent.

4.5.2 Alternatively, disposable plastic syringes with a barrel smaller than the neck of the soil vial may be used to collect the sample. The syringe end of the barrel is cut off prior to sampling. One syringe is needed for each sample aliquot to be collected.

4.5.3 Portable balance - For field use, capable of weighing to 0.01 g.

4.5.4 Balance weights - Balances employed in the field should be checked against an appropriate reference weight at least once daily, prior to weighing any samples, or as described in the sampling plan. The specific weights used will depend on the total weight of the sample container, sample, stirring bar, laboratory reagent grade water added, cap, and septum.

5.0 REAGENTS

5.1 Organic-free laboratory reagent grade water - All references to water in this method refer to organic-free laboratory reagent grade water.

5.2 Methanol, CH_3OH - purge-and-trap quality or equivalent. Store away from other solvents.

5.3 Sodium bisulfate, $NaHSO_4$ - ACS reagent grade or equivalent.

5.4 Polyethylene glycol (PEG), $H(OCH_2CH_2)_nOH$ – free of interferences at the detection limit of the target analytes.

5.5 See the determinative method for guidance on internal standards and surrogates to be employed in this procedure.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

The low concentration portion of this method employs sample vials that are filled and weighed in the field and never opened during the analytical process. As a result, sampling personnel should be equipped with a portable balance capable of weighing to 0.01 g.

6.1 Preparation of sample vials

The specific preparation procedures for sample vials depend on the expected concentration range of the sample, with separate preparation procedures for low concentration soil samples and high concentration soil and solid waste samples. Sample vials should be prepared in a fixed laboratory or other controlled environment, sealed, and shipped to the field location. Gloves should be worn during the preparation steps.

Because volatile organics will partition into the headspace of the vial from the aqueous solution and will be lost when the vial is opened, surrogates, matrix spikes, and internal standards should only be added to the vials back in the laboratory, either manually by puncturing the septum with a small-gauge needle or automatically by the sample introduction system, just prior to analysis.

6.1.1 Low concentration soil samples

Sodium bisulfate preservation is used in the preparation of vials used in the collection of low concentration soil samples to be analyzed by the closed-system purge-and-trap equipment described in Method 5035.

Water and subsequent freezing preparation of vials is used in the collection of low concentration soil samples known to contain carbonate minerals which may effervesce upon contact with an acidic preservation solution and which are to be analyzed by the closed-system purge-and-trap equipment described in Method 5035. This type of preservation is typically done in the lab after Encore samplers are received from the field. This must be done within 48 hours of sampling.

6.1.1.1 Add a clean magnetic stirring bar to each clean vial.

6.1.1.2 The preservative is added to each vial prior to shipping the vial to the field. Add 20 mL of 20% sodium bisulfate solution or 20 mL of water to the vial and seal the vial with the screw-cap and septum seal.

6.1.1.3 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible). It is important that labels and tape not cover the junction of the screw top and vial. Labels and tape must also be applied smoothly (i.e. no wrinkles) to prevent autosampler failures.

6.1.1.4 Weigh the prepared vial to the nearest 0.1 g and record it on the label.

6.1.2 High concentration soil samples in methanol:

6.1.2.1 When high concentration samples are collected without a preservative, a variety of sample containers may be employed, including 40-mL glass vials with septum seals (see Sec. 4.4).

6.1.2.2 The following steps apply to the preparation of vials used in the collection of high concentration soil samples to be preserved in the field with methanol and analyzed by the aqueous purge-and-trap equipment described in Method 5030.

6.1.2.3 Add 5 mL of methanol to each vial.

6.1.2.4 Seal the vial with the screw-cap and septum seal.

6.1.2.5 Affix a label to each vial. This eliminates the need to label the vials in the field and assures that the tare weight of the vial includes the label. (The weight of any markings added to the label in the field is negligible).

6.1.2.6 Weigh the prepared vial to the nearest 0.01 g and record it on the label.

NOTE: Vials containing methanol should be weighed a second time on the day that they are to be used. Vials found to have lost methanol (reduction in weight of >0.01 g) should not be used for sample collection.

6.1.3 Oily waste samples

When oily waste samples are known to be soluble in methanol, sample vials may be prepared as described in Sec. 6.1.2.2, using the appropriate solvent. However, when the solubility of the waste is unknown, the sample should be collected without the use of a preservative, in a vial such as that described in Sec. 6.1.2.1.

6.2 Sample collection

Collect the sample according to the procedures outlined in the sampling plan. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of the volatile components. Several techniques may be used to transfer a sample to the relatively narrow opening of the low concentration soil vial. These include devices such as the EnCore™ sampler, the Purge-and-Trap Soil Sampler™ , and a cut plastic syringe. Always wear gloves whenever handling the tared sample vials.

6.3 Sample handling and shipment

All samples for volatiles analysis should be cooled to approximately 4°C, packed in appropriate containers, and shipped to the laboratory on ice, as described in the sampling plan. Samples should be shipped on the day of sampling if at all possible.

6.4 Sample storage

6.4.1 Once in the laboratory, store samples at 4°C until analysis. The sample storage area should be free of organic solvent vapors.

6.4.2 All samples should be analyzed as soon as practical, and within the designated holding time from collection. Samples not analyzed within the designated holding time must be noted and the data are considered minimum values.

6.4.3 When the low concentration samples are strongly alkaline or highly calcareous in nature, the sodium bisulfate preservative solution may not be strong enough to reduce the pH of the soil/water solution to below 2. Therefore, when low concentration soils to be sampled are known or suspected to be strongly alkaline or highly calcareous, additional steps may be required to preserve the samples. Such steps include: addition of larger amounts of the sodium bisulfate preservative to non-calcareous samples, or the addition of 5 mL of water and storage at -10° (taking care not to fill the vials so full that the expansion of the water in the vial breaks the vial), or significantly reducing the maximum holding time for low concentration soil samples.

7.0 PROCEDURES

This section describes procedures for the low concentration soil method, the high concentration soil method, and the procedure for oily waste samples. High concentration samples are to be introduced into the GC system using Method 5030. Oily waste samples

are to be introduced into the GC system using Method 5030 if they are soluble in a water-miscible solvent, or using Method 3585 if they are not.

For the high concentration soil and oily waste samples, the surrogate compounds may either be spiked into the solvent at the time of extraction or the laboratory reagent grade water containing an aliquot of the extract prior to analysis.

Please refer to Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique”, current revision, for more information on subsampling.

7.1 Low concentration soil method (Approximate concentration range of 5 to 200 µg/kg - the concentration range is dependent upon the determinative method and the sensitivity of each analyte.)

7.1.1 Purge and Trap Autosampler Operation

Prior to using this introduction technique for any GC or GC/MS method, the system must be calibrated by the analytical method to be used. When a GC/MS method is used, internal standard calibration is employed.

7.1.1.1 Establish the purge-and-trap instrument operating conditions. Adjust the instrument to inject 10 mL of water, to heat the sample to 40°C, and to hold the sample at 40°C for 1.5 minutes before commencing the purge process, or as recommended by the instrument manufacturer.

7.1.1.2 Carry out the purge-and-trap procedure as outlined in Secs. 7.1.2. to 7.1.4.

7.1.2 Sample purge-and-trap

This method is designed for a 5-g sample size, but smaller sample sizes may be used. The soil vial is hermetically sealed at the sampling site, and MUST remain sealed in order to guarantee the integrity of the sample. Gloves must be worn when handling the sample vial since the vial has been tared. If any soil is noted on the vial or cap, it must be carefully removed prior to weighing. Weigh the vial and contents to the nearest 0.01 g, even if the sample weight was determined in the field, and record this weight. This second weighing provides a check on the field sampling procedures and provides additional assurance that the reported sample weight is accurate. Data users should be advised on significant discrepancies between the field and laboratory weights.

7.1.2.1 Remove the sample vial from storage and allow it to warm to room temperature. Shake the vial gently, to ensure that the contents move freely and that stirring will be effective. Place the sample vial in the instrument carousel according to the manufacturer's instructions.

7.1.2.2 Without disturbing the hermetic seal on the sample vial, add 10 mL of organic-free laboratory reagent grade water, the internal standards, and the surrogate compounds. This is carried out either manually or using the automated sampler. Other volumes of organic-free laboratory reagent grade water may be used. However, it is imperative that all samples, blanks, and calibration standards have exactly the same final volume of organic-free laboratory reagent grade water. Prior to purging, heat the sample vial to 40°C for 1.5 minutes, or as described by the manufacturer.

7.1.2.3 For the sample selected for matrix spiking, add the matrix spiking solution described in Sec. 5.0 of Method 5000, either manually, or automatically, following the manufacturer's instructions.

7.1.2.4 Purge the sample with helium or another inert gas at a flow rate of up to 40 mL/minute (the flow rate may vary from 20 to 40 mL/min, depending on the target analyte group) for 11 minutes while the sample is being agitated with the magnetic stirring bar or other mechanical means. The purged analytes are allowed to flow out of the vial through a transfer line to a trap packed with suitable sorbent materials.

7.1.3 Sample Desorption

7.1.3.1 Non-cryogenic interface - After the 11 minute purge, place the purge-and-trap system in the desorb mode and preheat the trap to 245°C without a flow of desorption gas. Start the flow of desorption gas at 10 mL/minute for about four minutes. Begin the temperature program of the gas chromatograph and start data acquisition.

7.1.4 Trap Reconditioning

After desorbing the sample for 1 to 4 minutes, recondition the trap by returning the purge-and-trap system to the purge mode. Maintain the trap temperature at 245°C (or other temperature recommended by the manufacturer of the trap packing materials). After approximately 10 minutes, turn off the trap heater and halt the purge flow through the trap. When the trap is cool, the next sample can be analyzed.

7.2 High concentration method for soil samples with concentrations generally greater than 200 µg/kg.

The high concentration method for soil is based on a solvent extraction. A solid sample is either extracted or diluted, depending on sample solubility in a water-miscible solvent. An aliquot of the extract is added to organic-free laboratory reagent grade water containing surrogates, internal and matrix spiking standards (added manually or by the autosampler), purged according to Method 5030, and analyzed by an appropriate determinative method. The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below, beginning at Sec. 7.2.1. If solvent preservation was employed in the field, then the preparation begins with Sec. 7.2.4.

7.2.1 When the high concentration sample is not preserved in the field, the sample consists of the entire contents of the sample container. Do not discard any supernatant liquids. Remove a representative aliquot with a spatula.

7.2.2 For soil and solid waste samples that are soluble in methanol, add 5.0 g (wet weight) of sample to a tared 40-mL VOA vial using a calibrated (refer to Katahdin SOP, CA-102, Balance Calibration) top loading balance. Record the weight to 0.1 g. Add 5 mL of methanol to the vial containing the sample and shake for two minutes.

NOTE: The steps in Secs. 7.2.1, 7.2.2, and 7.2.3 must be performed rapidly and without interruption to avoid loss of volatile organics. These steps must be performed in a laboratory free from solvent fumes.

7.2.3 For soil and solid waste samples that were collected in methanol or PEG, weigh the vial to 0.01 g as a check on the weight recorded in the field.

7.2.4 For each new lot of methanol, add an appropriate aliquot of the methanol to 20 mL of organic-free laboratory reagent grade water and analyze by Method 5030 in conjunction with the appropriate determinative method. Proceed to Sec. 7.0 in Method 5030 and follow the procedure for purging high concentration samples.

7.2 High concentration method for oily waste samples

This procedure for the analysis of oily waste samples involves the dilution of the sample in methanol or PEG. However, care must be taken to avoid introducing any of the floating oil layer into the instrument. A portion of the diluted sample is then added to 5.0 mL of organic-free laboratory reagent grade water, purged according to Method 5030, and analyzed using an appropriate determinative method.

The specific sample preparation steps depend on whether or not the sample was preserved in the field. Samples that were not preserved in the field are prepared using the steps below, beginning at Sec. 7.3.2. If methanol preservation was employed in the field, then the preparation begins with Sec. 7.3.4.

7.3.1 For oily samples that are not soluble in methanol or PEG (including those samples consisting primarily of petroleum or coking waste), dilute or extract with hexadecane and shake for two minutes.

7.3.2 For oily samples that are soluble in methanol if the waste was not preserved in the field, tare a 10-mL volumetric flask, or a VOA vial, weigh 1 g (wet weight) of the sample into the tared vessel and add 10.0 mL methanol or PEG with a calibrated syringe. If a vial is used instead of a volumetric flask, it must be calibrated prior to use. This operation must be performed prior to opening the sample vial and weighing out the aliquot for analysis. Invert the vial a minimum of three times to mix the contents.

7.3.4 If the sample was collected in the field in a vial containing methanol or PEG, weigh the vial to 0.1 g as a check on the weight recorded in the field, and proceed with Sec. 7.3.5.

7.3.5 Regardless of how the sample was collected, the target analytes are extracted into the solvent along with the majority of the oily waste (i.e., some of the oil may still be floating on the surface). If oil is floating on the surface, transfer 1 to 2 mL of the extract to a clean GC vial using a Pasteur pipet. Ensure that no oil is transferred to the vial.

7.3.6 Add an appropriate aliquot of the methanol or PEG to 5.0 mL of organic-free laboratory reagent grade water and analyze by Method 5030 in conjunction with the appropriate determinative method. Proceed to Sec. 7.0 in Method 5030 and follow the procedure for purging oily waste samples.

7.4 Determination of % Dry Weight

If results are to be reported on a dry weight basis, it is necessary to determine the dry weight of the sample. Refer to Katahdin SOP, CA-717, for determination of % dry weight.

NOTE: It is highly recommended that the dry weight determination only be made after the analyst has determined that no sample aliquots will be taken from the 60-mL vial for high concentration analysis. This is to minimize loss of volatiles and to avoid sample contamination from the laboratory atmosphere. There is no holding time associated with the dry weight determination. Thus, this determination can be made

any time prior to reporting the sample results, as long as the vial containing the additional sample has remained sealed and properly stored.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 Before processing any samples, the analyst should demonstrate through the analysis of an organic-free laboratory reagent grade water method blank that all glassware and reagents are interference free. Each time a set of samples is extracted, or there is a change in reagents, a method blank should be processed as a safeguard against chronic laboratory contamination. The blank samples should be carried through all stages of the sample preparation and measurement.

8.2 Initial Demonstration of Proficiency - Each laboratory must demonstrate initial proficiency with each sample preparation and determinative method combination it utilizes, by generating data of acceptable accuracy and precision for target analytes in a clean matrix. The laboratory must also repeat this demonstration whenever new staff are trained or significant changes in instrumentation are made.

8.3 Sample Quality Control for Preparation and Analysis - See the appropriate analytical method to follow to demonstrate acceptable continuing performance on each set of samples to be analyzed. These include the method blank, either a matrix spike/matrix spike duplicate or a matrix spike and duplicate sample analysis, a laboratory control sample (LCS), and the addition of surrogates to each sample and QC sample.

9.0 METHOD PERFORMANCE

Refer to appropriate analytical method.

10.0 APPLICABLE DOCUMENTS/REFERENCES

“Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods,” Method 5035, SW-846, USEPA, Revision III, June, 1997.

“Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods,” Method 5035A, SW-846, USEPA, Revision III, June, 1997.

“Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods,” Method 3585, SW-846, USEPA, Revision IIIB, Nov., 2004.

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Current Version.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

LIST OF TABLES AND FIGURES

TITLE: **CLOSED-SYSTEM PURGE-AND-TRAP AND EXTRACTION FOR VOLATILE ORGANICS IN SOIL AND WASTE SAMPLES USING SW846 METHOD 5035**

TABLE 1

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-214-06	METHOD 5035, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures	(1) Use methanol prep for all high concentration soils. (2) For high concentration soils, leave all extract in the vial with the soil for storage.	(1) For high concentration soils from an unknown source, perform a solubility test. (2) For high concentration soils, pipet approximately 1 mL of extract into a GC vial for storage.

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-202
Revision History
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Page 1

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

Prepared By: GC/MS Group Date: 2/97

Approved By:

Group Supervisor: [signature] Date: 011201

Operations Manager: [signature] Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1.23.01

General Manager: [signature] Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03 8260B	Format changes, added pollution prevention, changes to calibration section, new limits, added instrument. Other minor changes throughout.	DN	1.23.01	1.23.01
04 8260B	Revised sections 7.5.3.1, 7.5.5, 7.7.1, 7.8.2 + Table 2 to comply with South Carolina. Added NH oxygenates to calibration.	DN	5.23.01	5.23.01
05 8260B	Updated VOA calibration standard mixes. Added statistical limits for LCS/MS/MSD recoveries and the updated corrective actions	DN	5.21.02	5.21.02
06 8260B	Reorganization of sections 4, 5, 6 and 7, and Tables and Figures. Added definitions and information for the new data processing system.	MRC	05.03.04	05.03.04
07 8260B	minor changes rewording of sect. 7.6.3 preservation of Calcareous soils	LAD	020305	020305

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
08 8260B	Added references, setup and operation for the Encon / Centurion autosampler / Purge and Trap. Added ref. to instrument "T" and removed instrument "Q". Edited Std. conc. to reflect new instrumentation. Minor changes throughout to reflect current practice and correct typos.	LAD	04/06	04/06
09 8260B	Sect. 4H - added list of waste streams generated and location of Safe Limits. Clarified RT window studies. Added reference to MI SOP. Removed Grand mean Calibration model. Added wording for project specific acceptance criteria. Added LCS marginal outlier criteria. Added wording clarifying Calibration verification Std. Criteria and corrective action. Reworded Correlation	LAD	LAD 7-25-07 ~~03/07~~ 07/07	~~03/07~~ 07/07
10	Coefficient criteria Updated Sections 7.4.5, 7.4.6, 7.4.7, 7.5.2, 8.1, 10.0 and Table 1 with DoD QSM version 4.1 criteria	LAD	08/09	08/09
11	Added Table 2 with DoD QSM V. 4.1 QC Requirements. Added if the MSD Batch requirement can not be fulfilled, a LCSD must be analyzed. Removed "2" instrument and added the "C" and "D" instruments.	LAD	04/10	04/10
LAD 05/11/11 12	Removed Tekmar 2000 and 2016 throughout. Sect. 7.3.1 - Removed ~~590~~ 5970 GC/MS instrument type. Sect. 7.4.7 - Added RRT information. Sect. 8.1 - Added S.C. marginal exceedence criteria. Sect. 9 - Added MDL, LOD and LOQ criteria. Updated figures.	LAD	05/11	05/11
13	Sect. 5 - Changed Cal Mix and ICV Std. Exp. from 7 to 14 days. Sect. 6 - Add Sample preservation info. Sect. 7.4.4.1 - Added S.C. exemption from 2nd order Cal. Sect. 7.5.1 - Added Extras mix to LCS. Sect. 7.6.12 - clarified noting why samples need to be reanalyzed. Sect. 8.1 - Added 10% rule for LCS, ICV and MS/D. Sect. 9 - Added LOD/LOQ definitions. Table 1 - Reworded CA	LAD	03/12	03/12
14	For ICV: Sect. 1 and 7 - Removed Quikform references and added reporting from KIMS. Sect. 7 - Removed Soil 200 ug/kg L level and added 80.3% level. Sect. 8 - Added additional marginal exceedance information. Throughout - Fixed typos and made minor edits.	LAD	04/13	04/13

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

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Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-202-14**, titled **ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260**.

Recipient: _____ Date: _____

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel to prepare and analyze aqueous and solid matrix samples for purgeable organics by GC/MS in accordance with SW-846 Method 8260, current revision.

This SOP will consolidate all aspects of the analyses in one working document, to be revised as necessary, for the purposes of consistency in data quality.

1.1 Definitions

VOC: Volatile Organic Compounds

VOA: Volatile Organic Analysis

ANALYTICAL BATCH: 20 or fewer samples that are analyzed together with the same method sequence and the same lots of reagents and with the handling practices common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): A quality control sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. Laboratory reagent grade water is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing a mid point standard. The calibration check verifies that instrument conditions are sufficiently similar to those at initial calibration.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

INDEPENDANT CALIBRATION STANDARD: A solution prepared from a stock standard solution independent of the standard that is used to calibrate the instrument. This is prepared as an LCS and analyzed after the calibration before any sample analysis.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control and to measure the degree of accuracy of the determination.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions containing target analytes are added to a sample matrix prior to sample

extraction, in the case of soils, and/or analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the spiked analytes. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single analyte or mix of certified standards, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition as well as extraction and chromatography characteristics, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate. Surrogates provide an indication of the accuracy for the analytical determination in a discrete sample matrix.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS) : A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of volatile organics by the current revision of EPA Method 8260. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training and Demonstration of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of volatile organics by Method 8260 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate logbook. Any deviations from the test or irregularities with the samples should also be recorded in the lab logbook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, partially-filled VOA vials and sample jars are returned to the appropriate refrigerators to be disposed of in adherence with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, Sample Disposal, current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP SD-903.

Sample aliquots used for analysis are disposed of in accordance with SOP SD-903 and the Katahdin Hazardous Waste Management Plan and Safety Manual. The soil samples must be decanted and the soil fraction disposed of separately in compliance with Katahdin's disposal policies.
There are three general types of waste generated while performing the 8260 method. The "K" waste is a combination of water, sample aliquot (post analysis), as well as internal and surrogate standards. "K" waste is generated when preparing QC, during sample analysis, and procedural cleanup. There are "K" satellites

attached to each GC/MS instrument as well as an additional satellite located adjacent to the VOA sample preparation bench. "O" waste consists of methanol (as well as trace amounts of volatile analytes) and is generated when standard preparation syringes are rinsed three times with methanol. The "O" waste stream satellite is located inside the fume hood. Organic soil waste stream "I" consists of any solid left over from sample preparation and/or analysis and is located inside the fume hood. All satellites listed above are stored in a secondary container and are located in the Volatile Organics Laboratory room 111.

2.0 SUMMARY OF METHOD

The general methodology involves purging aqueous and soil samples with helium, an inert gas, for a set period of time to efficiently transfer purgeable organics to the gaseous phase. Soil samples with higher contaminant levels are extracted with methanol prior to the helium purge. These volatile organics are then retained on a cooled trap (commercially available trap suitable for the methodology) before heating causes desorption into a gas chromatograph for compound separation. Detection occurs with an electron impact ionization mass spectrometer.

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of VOCs is analyzed immediately after a sample containing high concentrations of VOCs. During initial data review, all analyses are evaluated for potential carryover. Any samples that have suspected carryover are reanalyzed. GC/MS policy is to reanalyze a sample with positive detects greater than the Practical Quantitation Limit (PQL) that has been run immediately after a sample with the same positive detects over the upper limit of the calibration. Typically 2 or 3 rinsing blanks are analyzed at the end of a sequence. Samples are not analyzed on the instrument until a blank with no detects above PQL can be obtained. If the lines are determined to be contaminated, then the entire Tekmar or Archon must be backflushed with warm methanol and water.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 6890 & 5890

4.2 Mass Spectrometers (MS): HP5973, HP5972 and HP5970

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Columns: RTX-VMS, 40 meter, 0.18 mm ID or equivalent.

4.5 Purge and Traps: Archon 5100 and Centurion auto samplers, and Tekmar 3000 and Encon concentrators.

4.6 Purge tubes: 5 mL fritted and 25 mL fritted purge vessels and 40 mL VOA vials for soil analysis.

4.7 Hamilton Gastight syringes: 2.00 uL to 25.00 mL.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

5.0 REAGENTS

5.1 Purge and trap grade methanol

5.2 Organic-free Laboratory reagent grade water: Siemens, Poland Spring, or equivalent. This water may need to be purged with nitrogen to eliminate organic contaminants such as Methylene chloride and Chloroform, which are commonly found at ambient levels in the laboratory.

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”.

5.3.1 The expiration date for all standards is six months from date of opening the ampule with the following exceptions:

Volatile gases expire within 2 weeks of opening ampule (gases are dichlorodifluoromethane, chloromethane, bromomethane, vinyl chloride, chloroethane, and trichlorofluoromethane).

New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

5.3.2.1 Calibration Mix – Prepare a standard in purge and trap methanol containing the compounds listed below. The final concentration of each compound is 200 ug/mL (some individual analyte concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The

standard must be prepared every 14 days and stored in the VOA standards freezer between uses.

Acetone	Dibromochloromethane	P-Isopropyltoluene
Benzene	1,2-Dibromoethane	Methylene Chloride
Bromobenzene	Dibromomethane	4-Methyl-2-Pentanone
Bromochloromethane	1,2-Dichlorobenzene	Naphthalene
Bromodichloromethane	1,3-Dichlorobenzene	N-Propylbenzene
Bromoform	1,4-Dichlorobenzene	Styrene
Bromomethane	Dichlorodifluoromethane	1,1,1,2-Tetrachloroethane
2-Butanone	1,1-Dichloroethane	1,1,2,2-Tetrachloroethane
n-Butylbenzene	1,2-Dichloroethane	Tetrachloroethene
sec-Butylbenzene	1,1-Dichloroethene	Tetrahydrofuran
tert-Butylbenzene	cis-1,2-Dichloroethene	Toluene
Carbon Disulfide	Trans-1,2-Dichloroethene	1,2,3-Trichlorobenzene
Carbon Tetrachloride	1,2-Dichloropropane	1,2,4-Trichlorobenzene
Chlorobenzene	1,3-Dichloropropane	1,1,1-Trichloroethane
Chloroethane	2,2-Dichloropropane	1,1,2-Trichloroethane
2-Chloroethylvinyl Ether	1,1-Dichloropropene	Trichloroethene
Chloroform	Cis-1,3-Dichloropropene	Trichlorofluoromethane
Chloromethane	Trans-1,3-Dichloropropene	1,2,3-Trichloropropane
2-Chlorotoluene	Ethylbezene	1,2,4-Trimethylbenzene
4-Chlorotoluene	Hexachlorobutadiene	Vinyl Acetate
Cyclohexane	2-Hexanone	Vinyl Chloride
1,2-Dibromo-3-Chloropropane	Idomethane	1,3,5-Trimethylbenzene
Isopropylbenzene	Methyl Tert-Butyl Ether	1-Chlorohexane

5.3.2.2 Extras mix – Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 200 ug/mL. The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

Acetonitrile	Isobutyl Alcohol
Acrolein	Methacrylonitrile
Acrylonitrile	Methylcyclohexane
Allyl Chloride	Methyl Acetate
Chloroprene	Methyl Methacrylate
Diethyl Ether	Methyl Tert-Butyl Ether
Cis-1,4-Dichloro-2-Butene	Pentachloroethane
Trans-1,4-Dichloro-2-Butene	Propionitrile
1,4-Dioxane	Tertiary-Amyl Methyl Ether
Di-Isopropyl Ether	Tertiary-Butyl Alcohol
Ethyl Methacrylate	1,3,5-Trichlorobenzene
Ethyl Tertiary-Butyl Ether	1,2,3-Trimethylbenzene
Freon-113	

5.3.2.3 Independent Calibration Verification Standard, Laboratory Control Spike and MS/MSD Mixture - Prepare a standard as above containing the compounds listed in Table 3. The final concentration of each compound is 200 ug/mL (some individual analyte

concentrations may vary, i.e. Ketones). The standard should be prepared in a 1.0 mL conical vial with a mini-inert valve cap. The standard must be prepared every 14 days and stored in the VOA standards freezer between uses.

5.3.2.4 Surrogate Spiking Solution - Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 250 ug/mL or 50 ug/mL depending on which autosampler you will be using. The standard must be prepared every 14 days and stored on the Archon and/or the Centurion autosampler in a pressurized vial or in the VOA standards freezer between uses.

4-Bromofluorobenzene
1,2-Dichloroethane-D_4
Toluene-D_8
Dibromofluoromethane

5.3.2.5 Internal Standard Solution - Prepare a standard as above containing the compounds listed below. The final concentration of each compound is 250 ug/mL or 50 ug/mL depending on which autosampler you will be using. The standard must be prepared every 14 days and stored on the Archon and/or the Centurion autosampler in a pressurized vial or in the VOA standards freezer between uses.

Pentafluorobenzene
1,4-Difluorobenzene
Chlorobenzene-D_5
1,4-Dichlorobenzene-D_4

5.3.2.6 BFB Solution - Prepare a standard as above containing 4-BFB. The final concentration is 25 ug/mL. The standard must be prepared every 30 days and stored in the VOA standards freezer between uses.

NOTE: The concentrations of standards may vary depending on the type of autosampler being used.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Aqueous samples

All aqueous samples are collected in 40 mL VOA bottles with no headspace, preserved with 1:1 HCl to a pH of <2 and stored at <6°C until analysis. Aqueous samples must be analyzed within 14 days from sample collection if preserved and within 7 days from sample collection if unpreserved.

6.2 Soil Samples

Soil samples arriving at the laboratory in Terra-core or Encores Soil samplers must be extruded into water or sodium bisulfate within 48 hours of sampling. Soils samples extruded into water must be frozen at -15°C ± 5°C until analysis. Soil sample extruded into sodium bisulfate must be stored at <6 °C until analysis.

Medium level soil (methanol preserved) samples are sampled into pre-weighed vials containing 5 mLs methanol. Methanol preserved soil samples must be stored at <6°C from the time of receipt at the lab until analysis.

Bulk soil samples are stored at <6 °C until analysis.

All soil/sediments must be analyzed within 14 days from sample collection.

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 “Standard Preparation and Documentation”.

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition: C:\HPCHEM\1\DATA

Tune file: BFB.U

Method files:

For BFB Tune: BFB288AQ.M (waters) or BFB288SL.M (soils)

For all samples and standards: I826AXX.M

where: I = instrument ID (Each instrument is given a unique identifier).
A = matrix (A for water, S for soil and SB for sodium bisulfate soils)
XX = the calibration number in chronological order

Data files:

For BFB: IB_ _ _.D

where: I is the instrument ID
_ _ _ is a number in chronological order from 000 to 999.

For all other data files: I_ _ _ _.D

Where: I is the instrument ID
_ _ _ _ is a number in chronological order from 0000 to 9999.

This file also contains the Quantitation output file.

7.3 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks, or samples, the GC/MS system must be shown to meet the mass spectral ion abundance criteria for a 50 ng injection of p-Bromofluorobenzene (p-BFB), tabulated below:

Mass	Criteria
50	15.0-40.0% of mass 95
75	30.0-60% of mass 95
95	base peak, 100% relative abundance
96	5.0-9.0% of mass 95
173	less than 2.0% of mass 174
174	greater than 50.0% of mass 95
175	5.0-9.0% of mass 174
176	greater than 95.0%, but less than 101.0% of mass 174
177	5.0-9.0% of mass 176

7.3.1 The following are the GC/MS operating conditions for injection of BFB.

GC/MS type: 5972 and 5973

Column:	RTX-624, 40 meter, 0.18 mm I.D or RTX-VMS, 40 meter, 0.18 mm ID.
Temperatures: Injection port:	200°
Transfer line:	150°
Detector:	240°
Isothermal temperature:	150°
Run time:	8 minutes
Scan start time:	3 minutes
Scan parameters:	not to exceed 2 sec per scan
Mass range:	35-300
Number of A/D samples:	8
GC peak threshold:	1000 counts
Threshold:	10 counts

The BFB solution must be analyzed once at the beginning of each 12-hour period, the time stamp of the injection of the BFB is the beginning of the 12-hour clock. All calibrations and samples must be run within the 12-hour clock as the method specifies.

When the BFB run has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The BFB run is processed using the current algorithms in the Target software.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, BFB must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument BFB tune is not in criteria.

7.4 INSTRUMENT CONFIGURATION / CALIBRATION

7.4.1 Tekmar LSC 3000/Archon 5100, Setup/Operation: Please refer to the Tekmar or Archon Manuals for more detailed operations for these instruments.

To begin, set the Tekmar 3000 to the specification listed in section 2-12 of the Archon manual. Edit method 14 as follows:

Method 14 should include:

Standby:	35°
Prepurge:	0 min
Preheat Temp:	0°
Sample Temp:	0°
Purge:	11 min
Dry purge:	2-4 min
Desorb preheat:	245°
Desorb Temp:	250°
Desorb time:	2-5 min
Dry purge:	2-4 min
Bake Time:	10 min
Bake Temp:	260°
Auto drain:	On
Bake gas by pass:	Off
Valve Temp:	120°
Line Temp:	120°
Runs per sample:	1

The above temperature settings are for a Vocarb 3000 trap, these temperatures may vary with the use of alternative traps. Temperature settings may also vary to optimize system performance.

The Archon autosampler should be set up according to the specifications in the manual. The setting of particular concern, with regards to keeping the Tekmar and Archon in coordination with each other, is the desorb time. There are several other programmable features on the Archon; the settings for this feature will depend on the sample matrix and method of analysis. Please refer to the Archon manual for more specifics on its programming features.

7.4.2 Encon/Centurion, Setup/Operation

Please refer to the Encon or Centurion manuals for more detailed operations for the instruments.

To begin, the Encon operation method should contain:

Purge Conditions:	Purge Gas: Helium Purge Time: 11.0 ±0.1 minute Purge Flow Rate: approx. 24-40 mL/min Purge Temperature: Ambient (water)
Desorb Conditions:	DesorbTemp: 250ºC Desorb Flow rate: 15 mL/min Desorb Time: 2.0 ± 0.1 min Bake Time: 10 min Bake Temperature: 260ºC

The above temperature settings are for a Vocarb 3000 trap, these temperatures may vary with the use of alternative traps. Temperature settings may also vary to optimize system performance.

The Centurion autosampler should be set up according to the specifications in the manual.

7.4.3 Initial Calibration for Method 8260

Once the instrument has achieved BFB tuning criteria, calibration of the instrument can begin.

To determine the linearity of response, the GC/MS must be initially calibrated at six different levels.

For aqueous calibration, target analytes and surrogate are prepared at the following concentrations; 1.0, 5.0, 20, 50, 100 and 200 ug/L. The curve is analyzed at ambient temperature.

For a soil calibration target analytes and surrogates are prepped at the following concentrations: 5.0, 10, 20, 50, 80 and 100 ug/L. The calibration standards are stirred and heated to 40ºC.

The following amounts standards should be added to 100 mL of organic-free laboratory reagent grade water in order to generate a 6-point initial calibration curve:

Notes	STD. ID	CAL. Mix 200 ug/mL	Extras Mix 200 ug/mL	Surr. Mix 250 ug/mL Archon	Surr. Mix 200 ug/mL Centurion
AQ curve only	VSTD001	0.5 uL	0.5 uL	0.4 uL	0.5 uL
	VSTD005	2.5 uL	2.5 uL	2.0 uL	2.5 uL
SL curve only	VSTD010	5.0 uL	5.0 uL	4.0 uL	5.0 uL
	VSTD020	10 uL	10 uL	8.0 uL	10 uL
CCV	VSTD050	25 uL	25 uL	20 uL	25 uL
SL curve only	VSTD080	75 uL	75 uL	30 uL	75 uL
	VSTD100	50 uL	50 uL	40 uL	50 uL
AQ Curve only	VSTD200	100 uL	100 uL	80 uL	100 uL

The internal standard is spiked by the autosampler. Due to different spike amounts separate standards are used depending on which autosampler is being used.

After analysis of the six points, the standard analyses must be quantitated and evaluated for adherence to QC criteria, as follows. Minimum requirements for method files are use of specific quantitation ions and quantitating a specific set of target compound and surrogates with a specified internal standard. These requirements are found in Tables 3 and 5.

7.4.4 Initial Calibration Criteria

The percent (%) RSD for six calibration check compounds (CCC) must be less than or equal to 30%. CCCs are 1,1-Dichloroethene, Chloroform, 1,2-Dichloropropane, Toluene, Ethylbenzene, and Vinyl Chloride.

A system performance check must be performed as part of initial calibration. The five system performance check compounds (SPCC) and the minimum acceptable average relative response factors (RRF) for these compounds are as follows (taken from 8260B):

SPCC	RRF
Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

The SPCCs are used to check both the standard and instrument stability.

7.4.4.1 Linearity of Target Analytes

If the RSD of any target analyte is 15% or less using the average response factor, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15% using the average response factor, then a calibration option outlined in section 7.0 of method 8000 will need to be employed. Please note that some options may not be allowable for certain states, federal programs, or clients.

Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. For linear models, Target calculates the correlation coefficient and then squares it (r^2). This is what is reported on all Target forms. The value for r^2 must be greater than or equal to 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order (seven calibration points required) polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.99.

Note: Non-linear calibration model may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear calibration for compliance work originating in their state. In these cases, a linear calibration model must be used.

7.4.5 Independent Calibration Verification

Immediately following an initial calibration, an independent calibration standard must be analyzed. This standard contains all target compounds, internal standards and surrogates at a concentration of 50 ug/L and is obtained from a source independent of the initial calibration source. Please refer to section 8.1 and Table 1 for acceptance criteria and corrective action for this standard.

For projects or clients requiring DoD QSM, current revision, all project analytes must fall between 80-120% of the true value. No samples may be run until the ICV criteria are met.

7.4.6 Calibration Verification

Once a valid initial calibration curve has been achieved, a continuing calibration standard containing all the target compounds, internal standards and surrogates at a concentration of 50 ppb must be analyzed every 12-hour clock for Method 8260, timed from the injection of BFB. The relative response

factor from the 50 ppb continuing calibration check standard must be compared to the average response factor data from the initial calibration.

The EICP (extracted ion current profile) area for any of the internal standards in the calibration verification must not change by more than a factor of two (-50% to +100%) from the same level standard in the last initial calibration. The retention time for any internal standard cannot shift by more than 30 seconds from the same level standard in the last initial calibration.
For Method 8260, if the percent difference for each CCC is less than or equal to 20%, and all of the SPCCs have a relative response factor greater than or equal to those listed in Section 7.4.4, the continuing calibration is considered valid.

For projects or clients requiring DoD QSM, current version, all project analytes must have ± 20%D.

Continuing calibration check criteria must be met before sample analysis can proceed.

7.4.7 Retention Time Windows

Retention time windows for each analyte and surrogate are set at the midpoint standard of the calibration curve, following every ICAL. On days when an ICAL is not performed, the initial CCV may be used. For each sample, the RRT shall be compared with the mid-point of the ICAL or the most recently updated RRT. If the RRT has changed by more than ± 0.006 RRT units indicates a significant change in system performance and the laboratory must take appropriate corrective action.

7.5 QUALITY CONTROL SAMPLE ANALYSIS

When preparing standards in water or spiking samples with internal standards/surrogates or matrix spike solution, be sure to rinse all syringes a minimum of three times with purge and trap grade methanol between uses. Failure to do this will result in cross-contamination of samples and standards.

7.5.1 Laboratory Control Sample (LCS)

The LCS mix is prepared from a secondary source vendor (i.e. different vendor from the calibration standards). The LCS is analyzed immediately after the initial calibration curve or calibration check and prior to the method blank to minimize any analyte carryover possibilities in samples. Acceptance criteria for the LCS are outlined in Section 8.0.

To prepare the water and medium-level soil LCS, 25 uL of the LCS and Extras standard mix at 200 ug/mL are spiked into 100 mL of analyte-free laboratory reagent grade water for a final concentration of 50 ug/L. The Archon

autosampler adds 1 uL of internal and 1 uL of surrogate standard to a 5 mL aliquot of this preparation for analysis. The Centurion autosampler adds 5 uL of both surrogates and internal standards to a 5 mL aliquot. To prepare the low-level soil LCS, a stir bar is added to 5 mL of the above solution in a VOA vial. The Archon unit adds an additional 10 mL of water to which the internal and surrogate standards have been added; this preparation is then heated, stirred and purged.

NOTE: In the event that the batch MS/MSD requirement cannot be fulfilled, a laboratory Control Spike Duplicate must be analyzed.

7.5.2 Method Blank Analysis

After calibration criteria have been met, a method blank must be analyzed before sample analysis can proceed. A method blank analysis must be performed once for each 12-hour calibration immediately after analysis of the calibration standard(s) and prior to sample analysis.
The aqueous method blank is a volume of analyte free laboratory reagent grade water spiked with internal and surrogate standards.

The low-level soil method blank is a volume of analyte free laboratory reagent grade water spiked with internal and surrogate standards. This method blank is analyzed using the low soil specification.

The method blank must contain less than the Practical Quantitation Level (PQL) for all analytes of interest for the samples associated with the blank.

For projects requiring DoD QSM, current version, no analytes may be detected >1/2 the PQL and > than the $1/10^{th}$ the measured amount in any sample or $1/10^{th}$ the regulatory limit, whichever is larger. Except for common laboratory contaminants which may not be detected > than the PQL.

7.5.3 Surrogate Recovery Limits

Laboratory established limits are derived for each of the surrogates. Please refer to the current revision of Katahdin Analytical Services SOP # QA-808 for further information on statistical limits. All samples including blanks, laboratory control samples, matrix spikes and client samples, must meet the statistical limits for the analysis to be considered valid. If surrogate recoveries do not meet these limits, reanalysis must occur to confirm matrix interference.

7.5.4 Internal Standard Area Recoveries / Retention Times.

The internal standard responses and retention times in the method blank must be evaluated immediately after or during data acquisition. If the EICP (extracted ion current profile) area for any of the internal standard changes by

a factor of two (-50% to +100%), from the last daily calibration standard, the GC/MS must by inspected, and corrective action taken. If the retention time for any internal standard has shifted by more than 30 seconds from the mid-point standard level of the most recent calibration sequence, the GC/MS must be inspected, and corrective action taken. All samples and QC must also meet the EICP area and retention time criteria or must be reanalyzed.

For projects or clients requiring DoD QSM, current version, IS EICP areas must be within -50% to + 100% of the ICAL midpoint standard. The retention time must be ± 30 seconds from the retention time of the ICAL midpoint standard.

7.5.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analysis

An MS/MSD must be analyzed every twenty samples of a similar matrix. The MS/MSD is prepared in a manner similar to the LCS, except that 40 mL aliquots (aqueous) or 5 g aliquots (soil), of environmental samples are used in place of the analyte-free laboratory reagent grade water. Note that trip blanks and field/equipment blanks should not be used for MS/MSD analyses. The spike solution (section 7.5.1) is added to the sample at a concentration of 50 ppb. Acceptance criteria for the MS/MSD are outlined in Section 8.0.

NOTE: In the event that sufficient volume of sample is not supplied to the laboratory so that an MS/MSD set cannot be analyzed within a batch of 20 samples, a laboratory control spike duplicate must be analyzed.

7.6 SAMPLE ANALYSIS

When new samples are received, they should be checked for past sample history. If sample history cannot be located or the sites are different than past sites, the project manager should be consulted. He/she may be able to provide more information about the sample. Sample history is used to determine what order in which to run the samples and at what dilution. Refer to Katahdin Analytical Services SOPCA-106, “Basic Laboratory Technique”, current revision for information on subsampling.

Samples are removed from the VOA refrigerator and appropriate chain of custody form is completed. Remove only the vials that have not been opened yet (opened vials will be upside down). Note in sample run log any bubbles, and significant discoloration or sediment in the sample vials.

7.6.1 SAMPLE ANALYSIS FOR 8260B WATER

7.6.1.1 Tekmar LSC 3000 / Archon 5100 units

Place the sample vials into the Archon sample tray and program the Archon for the appropriate sample volume and or dilution for the

sample. The Archon unit will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standard. The Archon can be programmed to run as many samples as will fit in the twelve-hour window. The auto sampler hot water rinses the sparge vessel, transfer lines, purge needle, and syringe between samples to minimize possible carryover.
Record the sample pH in the injection logbook after sample analysis is complete (usually the day after the analysis is done) and return the sample vial to the sample refrigerator.

7.6.1.2 Centurion/Encon unit

Place the sample vials into the Centurion sample tray and program the Centurion for the proper sequence. The Centurion will automatically transfer the sample to the sparge vessel while adding the internal and surrogate standards. Using the Centurion software, the analyst can program the Centurion to run as many samples that will fit into a 12 hour clock. The autosampler uses hot water to rinse the sparge vessel, transfer lines, purge needle and sample needle to minimize carryover.

Record the sample pH in the injection logbook after sample analysis is complete (usually the day after the analysis is done) and return the sample vial to the sample refrigerator.

Make sure that all entries in the injection log have been made in a complete, neat, and legible manner. Corrections in any logbook must be crossed through with a single line, dated, initialed and have a written explanation or the applicable error code.

If for any reason a sample needs to be rerun, diluted or duplicated, it must be noted in the comments section of the injection logbook. Additional information may be needed to assure that any questions that arise during the review process can be answered.

To minimize carryover from samples that contain a target compound at a level exceeding the upper limit of the calibration curve, the following must be done: monitor samples analyzed after the contaminated sample as well as the next run of the contaminated sample in the same purge inlet for the target(s) in question; both must have levels <PQL.

7.6.2 ANALYSIS OF LOW-LEVEL SOIL SAMPLES

Method 5035 Closed System Purge & Trap procedure for low level soils (5 ug/Kg -200 ug/Kg)

Selecting the appropriate technique may depend on cleanup goals, confidence levels, and anticipated levels of contamination. Field sampling activities typically result in Encore or Encore-like devices being submitted to the lab. These devices must be extruded within 48 hours. It is the laboratory's standard policy to extrude soil samples into 5 mL of Laboratory reagent free laboratory reagent grade water that contains a magnetic stir bar. The sample is subsequently frozen until analysis within 14 days. Note that the sample must be extruded and frozen within 48 hours of sampling, until analysis can begin. This approach is preferred over extrusion into sodium bisulfate because it is believed that the sodium bisulfate reacts with calcium carbonate in highly calcareous soils causing effervescence and driving the volatile analytes out of solution. There is also anecdotal information to suggest that acetone may be generated when bisulfate preservation occurs. The Katahdin sample ID, extrusion date, and time are recorded in the GC/MS extrusion logbook. Please refer to the Katahdin method 5035 SOP, CA-214 for more detail.

In lieu of the use of Encore samplers, the lab may pre-weigh 40 mL VOA vials containing 5 mL of laboratory reagent grade water or a 20% sodium bisulfate solution and a magnetic stir bar and ship these to the field. The vial is assigned a vial specific number prior to shipment to the field. The vial and weight will be recorded with its vial specific number in the methanol soil logbook. If possible the field sampler should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field. When the lab receives the vials back from the field, the vials will be weighed and the weight recorded. The samples must be frozen within 48 hours of sampling, until analysis can begin.

The subsequent analysis is performed on a specially developed autosampler that heats, stirs, and purges the sample simultaneously without exposing the contents of the vial to the atmosphere. This procedure will help to minimize the loss of VOC's due to transport, handling, and analysis and may help minimize ambient lab contribution. The expected detection limits are consistent with the traditional low soil technique from method 5030. The Archon is programmed to heat each vial to 40°C during the purge time. Initiate purging for 11.0 minutes; the sample must be heated to 40°C ± 1°C before purging can begin. If you have questions concerning setting up the Tekmar or initiating a GC/MS batch run, consult the Organic Department Manager, or senior chemist within the group.

If the client does not require method 5035, method 5030 for analysis of low-level soils may be followed. In this case, the Archon units may be used for the preparative step.

7.6.2 ANALYSIS OF MEDIUM-LEVEL SOIL SAMPLES

Method 5030 Procedure for higher concentration soils (> 200 ug/Kg)

Higher concentration soils may be sampled as either a bulk sample or field preserved with a water miscible solvent such as methanol. If sampled in an Encore unit, the soil is extruded into methanol upon receipt at the lab.

Bulk Sample- A sample is placed in a glass jar or vial and returned to the lab for extraction and analysis. In this approach the lab takes an aliquot of soil and extracts with purge & trap grade methanol, a portion of the methanol is then analyzed for volatile analytes.

Calibrate the balance properly (See SOP CA-102) and note it in the appropriate logbook. Place 5.0 grams of thoroughly mixed, undecanted soil sample in a 40.0 mL vial. Add 5.0 mL reagent grade methanol. Shake for 2 minutes. Let stand for 3 minutes. Record extraction in soil prep logbook.

Methanol Field Preservation - A 5 gram sample is added to a VOA vial that has been previously charged with purge and trap grade methanol (the volume of methanol is dependent upon client request). The vial with methanol has been previously weighed in the lab and assigned a vial specific number prior to shipment to the field. The vial and methanol weight will be recorded with its vial specific number in the VOA vial prep logbook. If possible the field sampler should weigh the sealed vial to ensure that 5 +/- 0.5 grams of sample were added in the field. When the lab receives the vials back from the field, the vials will be weighed and the weight recorded. A portion of the methanol is then analyzed for volatile analytes.

For analysis on Archon or Centurion autosamplers, add 400 uL of the extract into 20 mL of organic-free laboratory reagent grade water (e.g., Poland Spring or equivalent). IS and SS is added by the Archon and/or Centurion autosampler for analysis. This will give an estimated calibration range between 500-10000 ug/Kg.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is performed by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed.

- Surrogate recoveries
- stability of internal standard responses
- LCS spike recoveries
- method blank acceptance
- chromatography
- target compound detection/quantitation / review for false positives

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed.

7.7.1.1 Chromatography

The chromatography should be examined for the presence or absence of any "ghost" peaks and can also be used as an indication of whether or not matrix interferences might be influencing surrogate recoveries and/or ISTD area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g., Surrogate recoveries) to determine the necessity of reanalyses.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. An “M” qualifier will automatically be printed on the quantitation report summary.
This manual integration package must then be submitted to the Organic Department Manager or his/her designee, who will review each manual integration.

For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, “Manual Integration”, current revision.

7.7.1.2 Target Compound Detection/Quantitation

The method files have been set up to error on the side of false positives, that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- all ions present in the standard mass spectra at a relative intensity > 25% must be present in the sample spectrum.
- the relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- ions greater than 25% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the response of the largest target compound hit in the upper half of the initial calibration range.

The GC/MS laboratory initial data review should be accomplished at the beginning of a work shift for the previous set of analyses. After the analyst has completed his or her initial data review, the data should immediately be forwarded to the Organic Department Manager, or his/her designee.

7.7.1.3 Tentatively Identified Compounds (TIC)

TIC's may be requested by certain clients for samples. Refer to SOP CA-207 "GC/MS Library Search and Quantitation".

7.7.2 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into Kims. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Independent Calibration Verification, LCS and MS/MSD Criteria

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, "Generation and Implementation of Statistical QC Limits and/or Control Charts," current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

The LCS recoveries for all analytes are evaluated. For non-DOD clients, the exceedances from the laboratory established limits or nominal limits must be less than ten percent of the client compound list. For DOD clients, all of the compounds of interest must fall within either Katahdin's statistically derived limits or the DOD QSM, current version, limits with the following sporadic exceedance allowances.

Number of Analytes	Number of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

Any LCS failure must be evaluated to determine if it is within the marginal exceedance limits. These are listed in Appendix 3 of the DoD QSM. They also can be calculated for our statistically derived limit by extending the limit from 3 to 4 standard deviations.

Additionally, the exceedances must be random. Any analyte failing 2 out of 3 consecutive LCS's is considered to be non-random and may indicate another problem.

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

Note: South Carolina does not allow for marginal exceedences for compliance work originating in their state.

The MS/MSD recoveries for all analytes are evaluated. If the LCS results are acceptable but the MS/MSD is not, narrate. If both the LCS and MS/MSD are unacceptable reprep the samples and QC.

For projects or clients requiring DoD QSM, current version, all project analytes in the ICV must fall between 80-120% of the true value. No samples may be run until the ICV criteria is met. Laboratory established recovery limits for LCS and MS/MSDs must be within 3 standard deviations of the mean LCS recovery. MS/MSD pairs must be run once per analytical/preparatory batch. RPDs must be less than or equal to 30% between MS and MSDs.

For analytes with no available DoD acceptance criteria, laboratory established limits shall be used.

8.2 Surrogate Recovery Criteria

Statistical limits are compiled annually for surrogate recoveries (archived in QA office). Statistical limits are only calculated when at least 30 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the Organic Department Manager, Laboratory Operations Manager and Quality Assurance Officer. The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 60-140% or 70-130% may be used for some projects or states.

8.3 QC Requirements

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Due to the 14-day hold time associated with this method, samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

Limits of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD’s must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the anaysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 8260 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 8260B.

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Current Version.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

LIST OF TABLES AND FIGURES

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 1

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using BFB	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.3 of this SOP	Retune instrument, and verify
Six-point calibration for all analytes	Initial calibration prior to sample analysis	SPCCs average RF ≥0.30, except chloromethane, 1,1-DCA and bromoform ≥0.10; RSD for RFs ≤ 30% for CCCs. Refer to section 7.4.3 also.	Repeat initial calibration
Independent Calibration Verification	Once, immediately following calibration	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.1 of this SOP for more information on allowable exceedances.	If the surrogate recoveries in the ICV are low but the target analytes are acceptable, narrate. If the ICV recovery is high but the sample results are <PQL, narrate. If the ICV is out but the batch LCS is in criteria, narrate.
Calibration verification	Once per each 12 hours, prior to sample analysis in absence of initial cal	SPCCs minimum RF ≥ 0.30, except chloromethane, 1,1-DCA and bromoform ≥ 0.10; RF for CCC analytes ≤ 20% (%D) of average initial multipoint RF	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
IS	During data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Method Blank	One per batch of 20 or fewer samples.	No analytes of interest detected > PQL with the exception of Methylene Chloride	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.

TABLE 1 (cont.)

QC REQUIREMENTS - VOLATILE ORGANICS, METHOD 8260

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
LCS	One per batch of 20 or fewer samples.	Statistically derived from lab data or nominal limits depending on the project. Refer to QA records for statistical limits. Nominal limits are used as default limits. See also section 8.1 of this SOP for more information on allowable exceedances. .	Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Surrogate spike	Every sample, control, standard and method blank	Statistically derived limits.	Reprep and reanalyze for confirmation of matrix interference when appropriate.
MS/MSD	One MS/MSD per every 20 samples.	Statistically derived from lab data or nominal limits depending on the project. Statistical limits are used as default limits. .	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL Studies, LOD and LOQ Verifications	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
Demonstrate ability to generate acceptable P & A using 4 replicate analyses of a QC check standard	Once per year for each analyst; 4 reps	All recoveries within method QC acceptance limits	Recalculate results; locate and fix problem; rerun P & A study for those analytes that did not meet criteria prior to sample analysis

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 2

DOD QSM QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. RSD for RFs for CCCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression r ≥ 0.995; Option 3: non-linear regression–coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

TABLE 2 (cont)

DOD QSM QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. %Difference/Drift for all target compounds and surrogates ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.

TABLE 2 (cont)

DOD QSM QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements. In-house CLs may not be greater than ± 3 times the standard deviation of the mean LCS recovery. A number of analytes may fall outside the CL but within marginal exceedance limit depending on the total number of analytes in the LCS.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Refer to Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix if sufficient sample is available.	For matrix evaluation, use LCS acceptance criteria.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix if sufficient sample is available.	MSD: For matrix evaluation, use LCS acceptance criteria. MS/MSD: RPD ≤ 30% .	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

TABLE 2 (cont)

DOD QSM QC Requirements

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	The laboratory shall use laboratory surrogate CLs or use DoD-generated surrogate CLs, if available depending on project requirements. .	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepped within hold time.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-202-14	METHOD 8260, current revision
Apparatus/Materials	None	
Reagents	None	
Sample preservation/ handling	Preserved samples analyzed within 14 days. Unpreserved samples analyzed within 7 days.	Preserved samples analyzed within 14 days. No criteria for unpreserved samples.
Procedures	(1) Use laboratory reagent grade water for low level soil calibration, method blanks, and laboratory control samples to minimize clogging of archon soil needles with sand. (2) Internal Standards- pentafluoro-benzene, 1,4-difluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4	(1) Use an aliquot of a clean (control) matrix similar to the sample matrix. (2) Recommended internal standards – fluorobenzene, chlorobenzene-d5, 1,4-dichloro-benzene-d4
QC - Spikes	None	
QC - LCS	None	
QC - Accuracy/Precision	PQL – Practical Quantitation Level – three to ten times the MDL.	EQL – Estimated Quantitation Level – five to ten times the MDL
QC - MDL	None	

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 4

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Acetone	43	58
Acetonitrile	41	40, 39
Acrolein	56	55, 58
Acrylonitrile	53	52, 51
Allyl Chloride	76	41, 39
Benzene	78	-
Bromobenzene	156	77, 158
Bromochloromethane	128	49, 130
Bromodichloromethane	83	85, 127
Bromoform	173	175, 254
Bromomethane	94	96
2-Butanone	43	72
n-Butylbenzene	91	92, 134
Sec-Butylbenzene	105	134
Tert-Butylbenzene	119	91, 134
Carbon Disulfide	76	78
Carbon Tetrachloride	117	119
Chlorobenzene	112	77, 114
Chloroethane	64	66
2-Chloroethylvinyl Ether	63	65, 106
Chloroform	83	85
Chloromethane	50	52
Chloroprene	53	88, 90
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
Cyclohexane	56	84, 60
1,2-Dibromo-3-Chloropropane	75	155, 157
Dibromochloromethane	129	127
1,2-Dibromoethane	107	109, 188
Dibromomethane	93	95, 174
Diethyl Ether	74	45, 59
1,2-Dichlorobenzene	146	111, 148
1,3-Dichlorobenzene	146	111, 148
1,4-Dichlorobenzene	146	111, 148
Dichlorodifluoromethane	85	87
1,1-Dichloroethane	63	65, 83
1,2-Dichloroethane	62	98
1,1-Dichloroethene	96	61, 63
Cis-1,2-Dichloroethene	96	61, 98
Trans-1,2-Dichloroethene	96	61, 98
1,2-Dichloropropane	63	112

TABLE 4 (cont.)

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,1-Dichloropropene	75	110, 77
Cis-1,3-Dichloropropene	75	77, 39
Trans-1,3-Dichloropropene	75	77, 39
Cis-1,4-Dichloro-2-butene	75	53, 77
Trans-1,4-Dichloro-2-butene	53	88, 75
1,4-Dioxane	88	58, 43
Di-Isopropyl Ether	45	43, 87
Ethylbezene	91	106
Ethyl Methacrylate	69	41, 99
Ethyl Tertiary-Butyl Ether	59	87, 57
Freon-113	151	101
Hexachlorobutadiene	225	223, 227
2-Hexanone	43	58, 57, 100
Idomethane	142	127, 141
Isobutyl Alcohol	43	41, 42
Isopropylbezene	105	120
P-ISOPROPYLTOLUENE	119	134, 91
Methacrylonitrile	41	67, 39
Methylcyclohexane	83	55, 98
Methylene Chloride	84	86, 49
Methyl Acetate	43	74
Methyl Methacrylate	69	41, 100
4-Methyl-2-Pentanone	43	58, 85, 100
Methyl Tert-Butyl Ether	73	57, 41
Naphthalene	128	-
Pentachloroethane	167	130, 132
Propionitrile	54	52, 55
N-PROPYLBENZENE	91	120
Styrene	104	78
Tertiary-Amyl Methyl Ether	73	55, 87, 71
Tertiary-Butyl Alcohol	59	41, 43
1,1,1,2-Tetrachloroethane	131	133, 119
1,1,2,2-Tetrachloroethane	83	131, 85
Tetrachloroethene	164	129, 131, 166
Tetrahydrofuran	42	72, 71
Toluene	92	91
1,2,3-Trichlorobenzene	180	182, 145
1,2,4-Trichlorobenzene	180	182, 145
1,3,5-Trichlorobenzene	180	182, 145
1,1,1-Trichloroethane	97	99, 61
1,1,2-Trichloroethane	83	97, 85
Trichloroethene	95	97, 130, 132

TABLE 4 (cont.)

VOA COMPOUNDS AND CHARACTERISTIC IONS

COMPOUND	1° ION	2° ION
Trichlorofluoromethane	151	101, 153
1,2,3-Trichloropropane	75	77
1,2,3-Trimethylbenzene	105	120
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl Acetate	43	86
Vinyl Chloride	62	64
Xylenes (Total)	106	91
1-Chlorohexane	91	55,43

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

TABLE 5

ANALYTE QUANTITATION AND INTERNAL STANDARDS

Pentafluorobenze	**1,4-Difluorobenzene**	**Chlorobenzene - d5**	**1,4-Dichlorobenzene - d4**
Dichlorodifluoromethane	1,2-Dichloroethane	1,3-Dichloropropane	1,1,2,2-Tetrachloroethane
Chloromethane	1,1-Dichloropropene	Tetrachloroethene	1,2,3-Trichloropropane
Bromomethane	Carbon tetrachloride	Dibromochloromethane	Isopropylbenzene
Vinyl chloride	Benzene	Chlorobenzene	Bromobenzene
Chloroethane	1,2-Dichloropropane	1,1,1,2-Tetrachloroethane	2-Chlorotoluene
Trichlorofluoromethane	Trichloroethene	Ethylbenzene	4-Chlorotoluene
Methylene Chloride	Dibromomethane	Xylenes (total)	1,3,5-Trimethylbenzene
Acetone	Bromodichloromethane	Bromoform	Tert-Butylbenzene
1,1-Dichloroethene	cis -1,3-Dichloropropene	Styrene	1,2,4-Trimethylbenzene
1,1-Dichloroethane	4-Methyl-2-pentanone	2-Hexanone	Sec-Butylbenzene
cis-1,2-Dichloroethene	Toluene-d8 (surr.)	Bromoform	1,3-Dichlorobenzene
trans-1,2-Dichloroethene	Toluene		P-Isopropyltoluene
Chloroform	trans-1,3-Dichloropropene		1,4-Dichlorobenzene
2,2-Dichloropropane	1,1,2-Trichloroethane		1,2-Dichlorobenzene
2-Butanone	1,2-Dibromoethane		N-Propylbenzene
Methyl-tert-butylether (MTBE)	Vinyl Acetate		1,2-Dibromo-3-chloropropane
Tetrahydrofuran	Methyl Methacrylate		1,2,4-Trichlorobenzene
Bromochloromethane	Ethyl Methacrylate		Naphthalene
1,1,1-Trichloroethane	1,4-Dioxane		Hexachlorobutadiene
Tertiary-butyl alcohol (TBA)	2-Chloroethylvinyl ether		1,2,3-Trichlorobenzene
Di-isopropyl ether (DIPE)	Bromofluorobenzene (surr.)		cis-1,4-Dichloro-2-butene
Ethyl-tert-butylether (ETBE)			trans-1,4-Dichloro-2-butene
Tertiary-amyl methyl ether			Pentachloroethane
Diethyl Ether			n-Butylbenzene
Carbon Disulfide			1,3,5-Trichlorobenzene
Freon-113			1,2,3-Trimethylbenzene
Iodomethane			
Acrolein			
Isobutyl Alcohol			
Allyl Chloride			
Chloroprene			
Propionitrile			
Methacrylonitrile			
Acrylonitrile			
Cyclohexane			
Methyl Acetate			
Methylcyclohexane			
1-Chlorohexane			
Dibromofluoromethane (surr.)			
1,2-Dichloroethane-d4 (surr.)			

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 1

EXAMPLE OF VOA RUNLOG PAGE

KATAHDIN ANALYTICAL SERVICES

GCMS-D INSTRUMENT RUNLOG

DATE/TIME OF BFB INJECTION: 050611

SAMPLE NAME	DATAFILE	DF	ALS #	METHOD	PREP METHOD 5030	5035	1311	Criteria KAS	DoD	QAPP	Y/N	ANALYST	PH	COMMENTS
50 ng BFB	DB653	—	—	VOABFBAQ							Y	TTC	N/A	
VSTD050ADOGA	D1156	1	1	D8260A.sq							Y			
LCSA W691192-1	D1157	1	2								Y			
VBLKA	D1158	1	3								N			
VBLKB W691192-2	D1159	1	4								y			
SE2277-2RA B	D1160	1	5		X			X			y		<2	
SE2166-3RA B	D1161	1	6								y		<2	
SE2296-1 A	D1162	1	7								y		<2	
-2 A	D1163	1	8								y		<2	
-3 A	D1164	1	9								y		<2	
-4 A	D1165	1	10								y		<2	
-5 A	D1166	1	11								y		<2	
-6 A	D1167	1	12								y		<2	
-7 A	D1168	1	13								y		<2	
-8 A	D1169	1	14								y		<2	
SE2284-1 A	D1170	1	15								y		<2	
-2 A	D1171	1	16								y		<2	
SE2330-1 A	D1172	1	17								y		<2	DL 1:10
-2 A	D1173	1	18								y		<2	
-3 A	D1174	1	19								N		<2	RA co?
-4 A	D1175	1	20								y		<2	DL 1:4
Rinse	D1176	1	21								-		-	
	D1177	1	22								-		-	
													P 050611	

STANDARD	CODE
BFB	V3687
CAL. STD.	V3695/V3694
LCS/MS MIX	V3698
EXTRAS MIX	V3685

STANDARD	CODE
IS MIX	V3684
SS MIX	V3697

Circle Methods:

SW846 8260	OLM 04.2
SW846 8260 SIM	OLC 03.2
SW846 8260 SIM (heated purge)	EPA 624
	EPA 524

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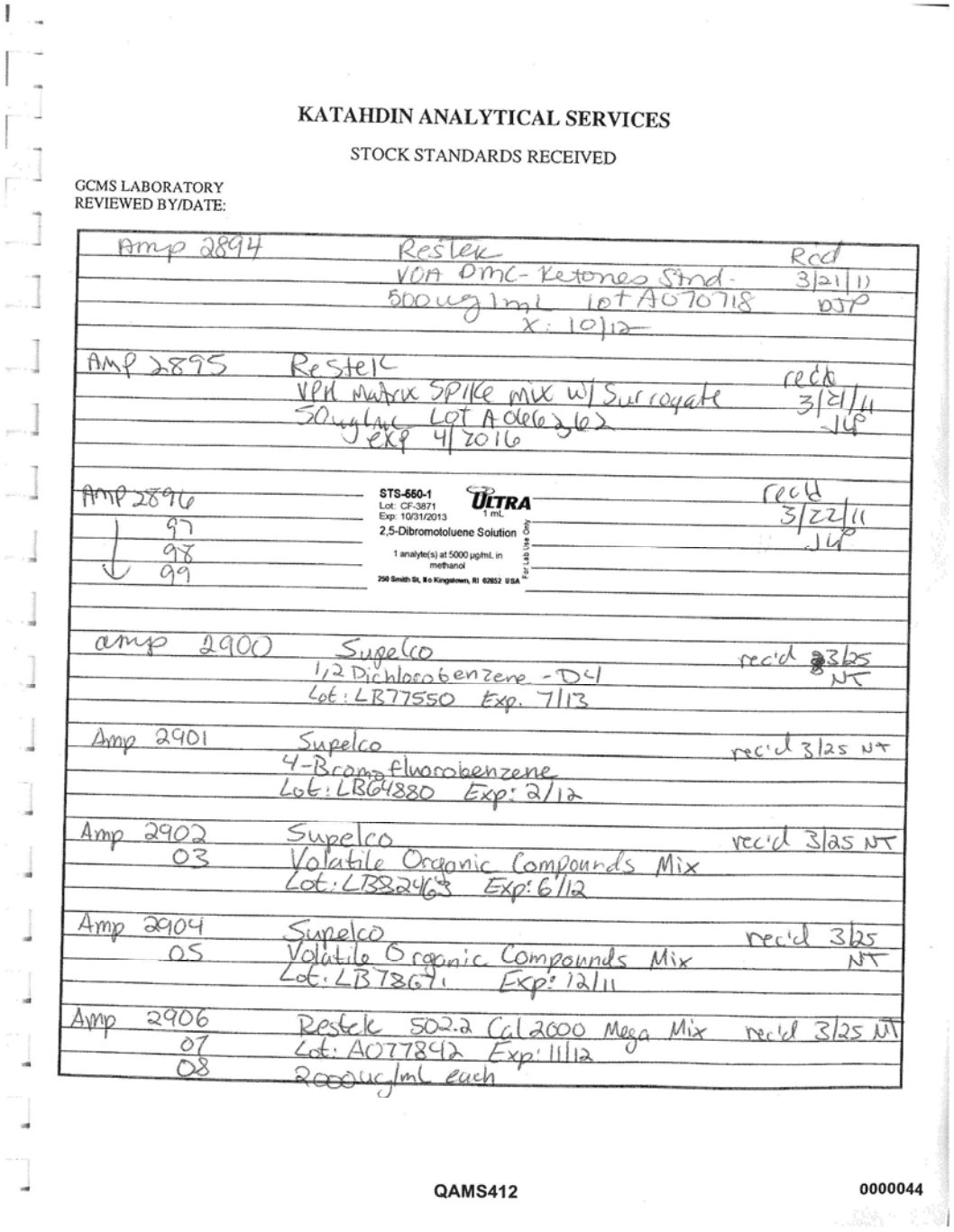
QAMS457

0000096

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS LABORATORY
REVIEWED BY/DATE:

Amp 2894 — Restek VOA DMC-Ketones Stnd. 500ug/mL lot A070718 X: 10/12 — Rcd 3/21/11 DJP

Amp 2895 — Restek VPH Matrix SPIKE mix W/ Surrogate 50ug/mL Lot A0606362 exp 4/2016 — rec'd 3/21/11 JLP

Amp 2896 97 98 99 — STS-550-1 Lot: CF-3871 Exp: 10/31/2013 ULTRA 1 mL 2,5-Dibromotoluene Solution 1 analyte(s) at 5000 µg/mL in methanol 250 Smith St, N Kingstown, RI 02852 USA — rec'd 3/22/11 JLP

amp 2900 — Supelco 1,2 Dichlorobenzene-D4 Lot: LB77550 Exp. 7/13 — rec'd 3/25 NT

Amp 2901 — Supelco 4-Bromofluorobenzene Lot: LB64880 Exp: 2/12 — rec'd 3/25 NT

Amp 2902 03 — Supelco Volatile Organic Compounds Mix Lot: LB82463 Exp: 6/12 — rec'd 3/25 NT

Amp 2904 05 — Supelco Volatile Organic Compounds Mix Lot: LB78671 Exp: 12/11 — rec'd 3/25 NT

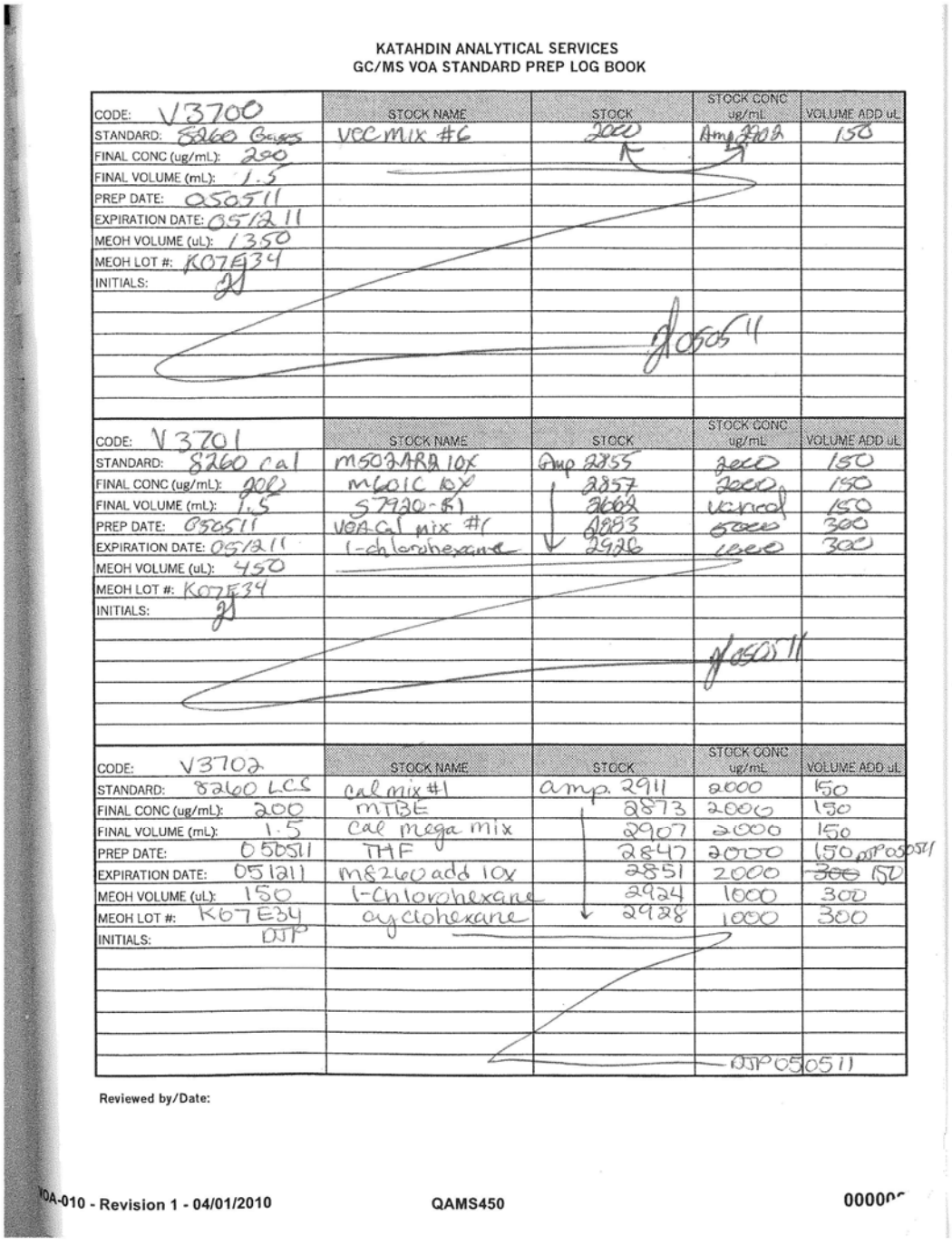
Amp 2906 07 08 — Restek 502.2 Cal 2000 Mega Mix Lot: A077842 Exp: 11/12 2000ug/mL each — rec'd 3/25 NT

QAMS412 0000044

TITLE: ANALYSIS OF VOAs BY PURGE AND TRAP GC/MS: SW-846 METHOD 8260

FIGURE 3

EXAMPLE OF VOA STANDARDS PREPARATION LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES
GC/MS VOA STANDARD PREP LOG BOOK

CODE: V3700	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 8260 Comps	VCC Mix #6	2000	Amp 2708	150
FINAL CONC (ug/mL): 200				
FINAL VOLUME (mL): 1.5				
PREP DATE: 050511				
EXPIRATION DATE: 051211				
MEOH VOLUME (uL): 1350				
MEOH LOT #: K07E34				
INITIALS: JJ				

CODE: V3701	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 8260 cal	MS02ARA 10X	Amp 2855	2000	150
FINAL CONC (ug/mL): 200	MS01C 10X	2857	2000	150
FINAL VOLUME (mL): 1.5	S7920-81	2609	various	150
PREP DATE: 050511	VOA Cal mix #1	2883	5000	300
EXPIRATION DATE: 051211	1-chlorohexane	2926	1000	300
MEOH VOLUME (uL): 450				
MEOH LOT #: K07E34				
INITIALS: JJ				

CODE: V3702	STOCK NAME	STOCK	STOCK CONC ug/mL	VOLUME ADD uL
STANDARD: 8260 LCS	cal mix #1	amp. 2911	2000	150
FINAL CONC (ug/mL): 200	MTBE	2873	2000	150
FINAL VOLUME (mL): 1.5	cal mega mix	2907	2000	150
PREP DATE: 050511	THF	2847	2000	150 DJP050511
EXPIRATION DATE: 051211	MS260 add 10x	2851	2000	~~300~~ 150
MEOH VOLUME (uL): 150	1-Chlorohexane	2924	1000	300
MEOH LOT #: K07E34	cyclohexane	2928	1000	300
INITIALS: DJP				

DJP050511

Reviewed by/Date:

VOA-010 - Revision 1 - 04/01/2010 QAMS450 000

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) and DBCP (1,2-Dibromo-3-Chloropropane) IN WATER BY SW846 METHOD 8011

Prepared By: Peter Lemay Date: 2/97

Approved By:

Group Supervisor: Peter Lemay Date: 1/25/01

Operations Manager: Jol C. Burton Date: 1/26/01

QA Officer: Deborah J. Nadeau Date: 1.25.01

General Manager: Duncan F. Heyden Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8011	Format changes, added pollution prevention, minor changes to sections 7+8 and QA Table.	DN	1.25.01	1/25/01
02	7.9.3 Removed sentence indicating analyst must date and/ initial manual integration "m" qualifiers 7.9.4 Changed last sentence to indicate we report the higher of the concentrations from the two different channels Removed section 7.4.6 from SOP Added reference to section 7.4.5 to Table 2 noting that Katahdin does not perform	DN	4.9.02	4.9.02
03	Changed definitions to include Target. Sect. 7.3.2 - editted table to reflect current calib. Replaced Turbochrom w/ Target in Sect. 7.8. Removed paragraph 4+5 in sect. 7.9.3. Replaced reporting (Sect. 7.10) w/ current practices. minor formatting changes.	LAD	030705	030705
04	Replaced Turbochrom with Target procedures Added DBCM standard information. minor formatting changes	LAD	03/06	03/06
05	Added waste streams to sect. 1.4. Changed QC reference sample to 0.1 ug/mL. Changed LCS and QCS limits to 60-140%. Added MI information. Added Sodium Thiosulfate as preservative for Samples. Added check for residual chlorine. Clarified correlation coefficient and coefficient of Determination	LAD	08/07	08/07

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) and DBCP (1,2-Dibromo-3-Chloropropane) IN WATER BY SW846 METHOD 8011

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added definition. Revised sections 7, 8, 10 and Table 1 to comply with DoD QSM Version 4.1.	LAD	08/09	08/09
07	Minor edits to reflect current practice. Added Table 2 with DoD QSM QC Criteria. updated figures 2 and 3.	LAD	12/10	12/10
08	Changed 0.1g to 0.01g throughout. Changed 7-point calibration to 6 point calibration throughout. Changed concentrations of the MDL & QC stds to 0.05 and 0.25 respectively. Updated prep of these Stds. Changed CAR to NCR. Added Method Performance information. Updated figures. Added preparation logbook Figure.	LAD	03/12	03/12
09	Sect. 1 & 7 - Removed Quickforms and added reporting through KIMS. Figures 1 → 3 - updated.	LAD	05/13	05/13

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) AND DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document **SOP CA-319-09**, titled **EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) and DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-319-09**, titled **EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) AND DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011**

Recipient: _____ Date: _____

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) AND DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011

1.0 SCOPE AND APPLICATION

This SOP describes the procedures utilized by Katahdin Analytical Services, Inc. technical personnel to analyze drinking water and groundwater for EDB and DBCP using SW846 method 8011. This document describes the microextraction technique and the gas chromatographic procedures.

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; however a universal blank matrix does not exist for solid samples, and therefore, no matrix is used. The blank is taken through the appropriate steps of the process.

QUALITY CONTROL REFERENCE SAMPLE (QCS): A solution of method analytes of known concentrations that is used to fortify an aliquot of laboratory reagent grade water. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with external prepared test materials.

LABORATORY CONTROL SAMPLE (LCS) (QC Check Standard): An aliquot of laboratory reagent grade water to which known quantities of the method analytes added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

STANDARD CURVE (CALIBRATION CURVE): A calibration where aqueous calibration standards are prepared and processed in exactly the same manner as a sample. The calibration standards are extracted using the same process as the samples. Using procedural standard calibration compensates for any inefficiency in the processing procedure. The curve plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing or more method analytes purchased from a reputable commercial source. Stock standard solutions are used to prepare calibration standards.

PRIMARY DILUTION STANDARD SOLUTION: A solution of several analytes prepared in the laboratory from stock standard solutions and diluted as needed to prepare calibration solutions and other needed analyte solutions.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the primary dilution standard solution and stock standard solution, which is used to calibrate the instrument response with respect to analyte concentration.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

PE NELSON TURBOCHROM or HP Chemstation: data acquisition systems that are used to collect chromatographic data. The systems can also be used to archive raw data files.

TARGET: An Oracle database used to store and organize all Target data files.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of EDB, DBCP according to SW846 method 8011. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the analysis of EDB and DBCP according to SW846 Method 8011 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Health and Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard.

NOTE: 1,2-Dibromoethane and 1,2-Dibromo-3-chloroproane have been tentatively classified as known or suspected human or mammalian carcinogens. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Wastes generated during standards preparation are disposed of in the Mixed Flammable Waste (O). The extraction VOA vial waste is disposed of in the Sep Funnel Aq Waste (N-Low). After the extracts have been analyzed, the autosampler vials and any expired standard vials or ampules are disposed of in the Organic Vial Waste (P).

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) AND DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011

2.0 SUMMARY OF METHOD

2.1 Thirty-five mls of sample are extracted with 2 ml of hexane. Two uL of the extract are then injected into a gas chromatograph equipped with a linearized electron capture detector for separation and analysis. Aqueous matrix spikes are extracted and analyzed in an identical manner as the samples in order to compensate for possible extraction losses.

2.2 The extraction and analysis time is 15 to 30 minutes per sample depending upon the analytical conditions chosen.

2.3 Confirmatory evidence is obtained using a dissimilar column.

3.0 INTERFERENCES

Impurities contained in the extraction solvent can cause analytical difficulties. Contamination is often not due to the analytes of interest but other organochlorine compounds. EDB at low concentrations may be masked by very high concentrations of Dibromochloromethane (DBCM) a common chlorinated drinking water contaminant.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph: GC Hewlett Packard 6890 series connected to the Turbochrom or HP Chemstation data system, or equivalent.

4.2 Columns: Instruments are configured with a pre-column originating from the injection port which is connected to deactivated glass Y splitter that connects two different columns to two detectors. The most commonly used columns are: RTX-35 30M x 0.53 mm ID, RTX-5 30M x 0.53 MM ID, or RTX-1701 30M x 0.53 mm ID. Equivalent columns can be used.

4.3 Detectors: - Electron capture detectors (ECD).

4.4 Analytical top loading balance capable of weighing to 0.01g.

4.5 Volumetric flasks, class A: sizes as appropriate with the ground-glass stoppers.

4.6 Syringes: various sizes for preparing standards and injecting samples on the instrument.

4.7 Vials: various sizes and types including crimp tops.

4.8 Glass VOA vials, 40.0mL capacity

4.9 50mL glass graduated cylinder

4.10 Disposable Pasteur pipettes

4.11 Refrigerator for storage of extracts and standards.

5.0 REAGENTS

5.1 Tetrachlorometaxylene (TCMX) surrogate solution (1000 µg/mL) in methanol.

5.2 Dibromochloromethane (DBCM) solution (100 µg/mL) in methanol.

5.3 Stock standard solutions: Certified solutions purchased from suppliers like Restek or other acceptable retailers. Expiration dates are six months from date of opening vial or sooner if manufacturers date is less. Upon receipt, all standards are logged into the appropriate logbook with the date of receipt, expiration date, source, lot number, solvent and concentration of compounds.

5.4 Primary Dilution standards: Prepared through the dilution of the stock standards with methanol. Methanol solutions prepared from liquid analytes are stable for at least four weeks when stored at 4° C. Information is documented in a separate logbook.

5.5 Quality Control Reference Sample. A certified solution containing the compounds EDB and DBCP from a vendor other than that used for initial calibration.

5.6 Sodium chloride, reagent grade crystals.

5.7 Laboratory reagent grade water.

5.8 Hexane - Baker analyzed or equivalent.

5.9 Methanol - demonstrated to be free of contaminants.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are colleted in 40 mL VOA vials preserved with HCL and filled with no headspace. This will dechlorinate the samples. They are stored at 4 (±2) °C until times of extractions.

Samples must be extracted within 14 days from the date of sampling. Sample extracts must be analyzed within 24 hours of extraction.

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) AND DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011

7.0 PROCEDURES

7.1 INSTRUMENT CONDITIONS:

Refer to the instrument logbook for the current column and conditions.

Typical conditions are:

Makeup flow: 60 mL/min Ar/Methane or Nitrogen
Column flow: 4 mL/min Helium
Injector Temp: 150°C
Detector Temp: 300°C
Oven Ramp: 80(0.5) - (12°C/min) - 200(0) - 25°C/min - 270(0.7)
Run time: 14 min
Injection size: 2 uL

7.2 SAMPLE PREPARATION

7.2.1 Remove samples and standards from storage and allow them to reach room temperature.

7.2.2 For samples and field blanks contained in 40-mL bottles, remove the container cap. Discard a 5-mL volume using a transfer pipette. Replace the container cap and weigh the container with contents to the nearest 0.01g and record the weight in the preparation logbook (Figure 1). This will be used for subsequent sample volume determination.

7.2.3 For calibration standards, check standards, QC reference samples, and blanks, measure a 35 mL volume using a 50 mL graduated cylinder and transfer it to a 40 mL sample container.

7.3 CALIBRATION STANDARDS

7.3.1 Prepare the following primary dilution standards:

7.3.1.1 Two mixes of EDB and DBCP: one at a concentration of 0.07 and another at a concentration of 0.70 ng/uL in methanol.

7.3.1.2 One mix of EDB and DBCP at a concentration of 0.07 ng/uL in methanol from a source other than that used for the initial calibration.

7.3.1.3 A solution of the surrogate at a concentration of 0.70 ug/ml in methanol.

7.3.1.4 A solution of DBCM at a concentration of 0.70 ug/ml in methanol.

7.3.2 Prepare six calibration standards at the following concentrations by adding sufficient quantities of the primary dilution standards to seven 40-ml VOA vials:

Initial Surr. Vol (ul)	Initial Std. Vol. (ul)	Initial Std. Conc. (ug/ml)	Final Vol. (ml)	Final Surr. Conc. (ug/L)	Final Std. Conc. (ug/L)
25	25	0.070	35	0.50	0.05
37.5	50	0.070	35	0.75	0.10
50	125	0.070	35	1.0	0.25
62.5	250	0.070	35	1.25	0.50
125	50	0.70	35	2.5	1.0
250	125	0.70	35	5.0	2.5

7.3.3 Laboratory Control Sample (check standard) - Prepare at a concentration of 0.25 ug/L by adding 125 ul of the 0.07 ug/ml primary dilution standard containing EDB and DBCP from section 7.3.1.1 to a 40 ml VOA vial.

7.3.4 MDL check sample (Laboratory Control Sample for assessing the laboratory sensitivity) - Prepare at a concentration of 0.05 ug/L by adding 25 ul of the 0.07 ug/ml primary dilution standard containing EDB and DBCP from section 7.3.1.1 to a 40 ml VOA vial.

7.3.5 Quality Control Reference Sample – Prepare at a concentration of 0.25 ug/L by adding 125 ul of the 0.07 ug/ml alternate source dilution standard containing EDB and DBCP from section 7.3.1.2 to a 40 ml VOA vial.

7.3.6 DBCM check sample – Prepare at a concentration of 1.0 ug/L by adding 500 ul of the 0.07 ug/ml standard containing DBCM from section 7.3.1.4 to a 40 ml VOA vial.

7.4 EXTRACTION

7.4.1 Add 125 uL of TCMX surrogate solution (0.70ng/uL) to each method blank, QC reference sample and sample VOA vial. The effective concentration of TCMX is 2.5 ug/L.

7.4.2 Remove the container cap and add 6 g of NaCl to all the samples.

7.4.3 Recap the sample container and dissolve the NaCl by shaking by hand for about 20 seconds.

7.4.4 Remove the cap and using a gas-tight syringe, add 2.0 ml of hexane. Recap and shake vigorously by hand for 3 minutes. Allow the water and hexane phases to separate. If stored at this stage, keep the container upside down.

7.4.5 Remove the cap and carefully transfer a sufficient amount (0.5 -1.0 ml) of the hexane layer into a vial using a disposable glass pipette.

7.5 CALIBRATION

7.5.1 The GC system is calibrated using the external standard calibration procedure. Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. The Target system will calculate a peak height for all compounds. A calibration curve can be prepared in Target using the peak height against the concentration of the standard. A non-linear calibration applying a second order polynomial (quadratic fit) equation is used to prepare the curve. In order to be used for quantitative purposes, the Coefficient of Determination (r^2) must be greater than or equal to 0.990. The quadratic equation is:

$y = ax^2 + bx + c$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = the intercept

7.5.2 A non-linear calibration model may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear calibration work originating in their state. In these cases, a linear calibration model must be used. Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. The Target system will calculate a peak height for all compounds. A calibration curve can be prepared in Target using the peak height against the concentration of the standard.

7.5.2.1 Linear calibration using the average calibration factor

The calibration factor (CF) is calculated using the following formula:

$CF = A_s / C_s$

where: A_s = Peak area (or height) of the analyte or surrogate.
C_s = Concentration of the analyte or surrogate, in µg/L.

To evaluate the linearity of the initial calibration, calculate the mean CF, the standard deviation (SD), and the RSD.
If the RSD of the calibration factor is less than or equal to 20% over the calibration range, then linearity through the origin may be

assumed, and the average calibration or response factor may be used to determine sample concentrations.

7.5.2.2 Linear calibration using a least squares regression

$y = bx + c$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = the intercept

The analyst should not force the line through the origin, but have the intercept calculated from the five data points. In addition, do not include the origin (0,0) as a sixth calibration point. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995. The ICAL must be successful before any samples or other QC check samples can be analyzed.

7.5.1 An LCS must be analyzed after an initial calibration prior to any samples being analyzed.

7.6 RETENTION TIME WINDOWS

7.6.1 Three injections of all single component standard mixtures throughout the course of a 72-hour period.

7.6.2 The standard deviation of the three retention times is calculated for each single component standard.

7.6.3 Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window; however, the experience of the analyst should weight heavily in the interpretation of chromatograms.

7.6.4 Retention time windows are calculated for each standard on each GC column at method setup and after major maintenance, including whenever a new GC column is installed. The data is kept on file in the laboratory.

7.6.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being too narrow. By utilizing

these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive by carefully evaluating the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.

7.7 GAS CHROMATOGRAPHIC ANALYSIS

7.7.1 All instrument injections are performed using the direct injection technique with an autosampler set for 2-5 uL injection volumes.

7.7.2 Samples are analyzed in a set referred to as an analytical sequence. A typical sequence includes the following:

- Initial Calibration Standards
- Quality Control Reference Sample - 0.1ug/L
- MDL Check sample - 0.05ug/L
- DBCM Check sample - 1ug/L
- Method Blank
- Laboratory Control Sample (Check standard) - 0.25ug/L
- Samples
- Calibration Verification Standard

The sequence begins by calibrating the instrument with a six point calibration as listed in Section 7.3 followed by the Quality Control Sample and the MDL check sample. If the calibration curve and the QC samples meet the acceptance criteria listed in section 8.3 the analysis is continued with a method blank, LCS and sample extracts. If a calibration curve has been analyzed previously on a different day, the calibration can be verified by analyzing a mid-point calibration verification standard (CV). If the recovery for each analyte is between 70% and 130% of the expected value, this CV can be used to start the analytical sequence and samples can then be analyzed. A CV has to be analyzed at the end of the analytical sequence or every 12 hours, whichever is first. For clients or projects requiring DoD QSM, current version, the response for any analyte must not vary from the expected response by more than ± 20%, or a new calibration curve must be prepared for that analyte. If the CCV fails the above criteria, reanalyze all samples since the last successful calibration verification. If reanalysis cannot be performed, data must be qualified and explained in the narrative. Additionally, apply a Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification. If the CV fails, the instrument is checked for any obvious problems and maintenance is performed if deemed necessary.

Another CV is analyzed or the instrument is recalibrated and then samples are injected. All samples that were injected after the standard exceeding the criterion must be reinjected to avoid errors in quantitation, if the initial analysis indicated the presence of the specific target analyte that exceeded the criterion.

7.7.3 The center of the retention time window for each analyte and surrogate is established by using the absolute retention time for each analyte and surrogate from the daily opening calibration verification or initial calibration.

7.7.4 An analyte is tentatively identified when a peak retention time from a sample is close to the retention time of the calibration standard and the check standard. Confirmation is required on a dissimilar GC column. If the compound is detected on a second column and the quantitation agrees within ±40%, then the higher value is reported.

7.7.5 If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.

7.7.6 When a GC system is determined to be out of control because either a CV can not pass or a six point calibration does not meet the correlation coefficient criteria, instrument maintenance is likely necessary. Routine instrument maintenance may involve changing the septum, replacing the liner, clipping the pre-column, or replacing the column. This information is recorded in the instrument run log (Figure 2). When an instrument requires more severe maintenance like replacing the ECD or an electronic board, this information is written in the instrument maintenance logbook.

7.8 DETERMINATION OF SAMPLE VOLUME

For samples and field blanks, remove the cap from the sample container. Discard the remaining sample/hexane mixture. Shake off the remaining few drops using short, brisk wrist movements. Reweigh the empty container with the original cap, record the weight to the nearest 0.01 g in the preparation logbook. Calculate the weight of sample by subtracting the weight of the vial from the original weight of the vial plus sample. Record the difference to the nearest 0.01g in the preparation logbook.

7.9 CALCULATIONS

7.9.1 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration when the file is processed through the appropriate calibrated method.

7.9.2 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration as follows (waters):

Concentration (ug/L) = (C) (D) (35) / (Vs)

Where: C=concentration calculated by Target in ug/L
D=Instrument dilution
Vs=Volume of sample extracted in ml

7.10 DATA REVIEW

7.10.1 Initial Data Review

The analyst who ran the samples accomplishes the initial data review. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed in Target Review. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: manual integration.
- Target compound detection: quantitation, confirmation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next workday. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.10.

7.10.2 Surrogate recovery

All recoveries must meet the most recently laboratory established acceptance limits, which are listed on the GC Laboratory Surrogate Acceptance Limit sheet.

The sample is evaluated for the recovery of the surrogate. If the surrogate recovery is high and the sample contains less than the PQL for all target analytes, the data is narrated. If the surrogate recovery is low and may be attributable to matrix interference or a matrix effect, the data is narrated. If the surrogate recovery is low and the sample concentration is less than the PQL for all target analytes and there is no apparent matrix effect, reextract the sample.

For method blanks, if the recovery of the surrogate is low or high, and the blank does not contain any target analytes above the PQL, and the recovery of the surrogate in the sample(s) are acceptable, the data is narrated. If the recoveries in the blank are low and it does not contain any target analytes above the PQL, and the recoveries in the samples are acceptable but the sample contains one or more target analytes above the PQL, the sample may be reextracted.

For laboratory control samples (LCS), if the only discrepancy in the extraction batch is with the LCS, and the analyte spike recoveries are acceptable, the data is narrated. If the recoveries of the surrogate and the analyte spikes are low, the samples may need to be reextracted.

7.10.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), they are preformed in Target Review. A “m” qualifier will automatically be printed on the quantitation report summary. The analyst will date and initial the “m” on the quanitation report summary and assign a code that indicates the reason for the manual integration. Refer to Katahdin SOP QA-812 “Manual Integration on GC/MS, GC, HPLC and IC Datasystems” for more information.

7.10.4 Target Compound Detection

The chromatogram from channel A is evaluated with that from channel B. If a target analyte is present on both channels and the concentration is within the calibration range, and the quantitation from both chromatograms agrees within ±40%, the analyte is considered to be present in the sample. The higher of the two concentrations is reported.

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be undetected electronically in Target. The

possible scenarios are: if an analyte is present on one column but its concentration is below the PQL, if an analyte is present on one column but does not confirm on the other channel, if an analyte is present on both columns but the concentrations differ by more than 40%, or if an analyte is present but its retention time is ±0.04 minutes or more than the retention time of the analyte in the preceding CV.

If reporting data that has an RPD that is >40%, the data must be flagged with a "J" indicating that the result is an estimated value. Sometimes interference on one column (i.e. sulfur) will prevent a target analyte from detection and it is present on the conformational column. In this scenario, the result would be reported from one column and need to be "Q" flagged to indicate that it was not confirmed on a second column.

7.11 REPORTING

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into KIMS. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to a secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When the completed, the package is sent to the Department Manager for final review. A completed review checklist (figure 3) is provided with each package. The final data package from the Organics Department is then processed by the Data Management Department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below and refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager and/or Quality Assurance Officer may be consulted to evaluate data. Due to time constraints, samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

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In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 For each analytical batch (up to 20 samples), a method blank, a laboratory control sample (check standard), a matrix spike and matrix spike duplicate are analyzed. For every initial calibration curve that is prepared and analyzed, a DBCM check and a MDL check sample are analyzed. Once a week a QC Reference Sample is analyzed. They are carried through all stages of the sample preparation and analysis steps.

8.2 Spike concentrations: The LCS (check standard) and the MS/MSD are spiked at the same concentration. The spike concentrations are:

Compound	ug/L
1,2-Dibromoethane (EDB)	0.25
1,2-Dibromo-3–chloropropane (DBCP)	0.25

The QC Reference Sample is spiked at:

Compound	ug/L
1,2-Dibromoethane (EDB)	0.25
1,2-Dibromo-3–chloropropane (DBCP)	0.25

The spike concentration of the MDL check sample is 0.05 ug/L.

The surrogate spike concentration is:

Compound	ug/L
Tetrachloro-m-xylene (TCX)	2.5

8.3 QC Reference, LCS, and MS/MSD acceptance criteria and Corrective Action: All QC samples are calculated for percent recovery of the spiked analyte(s). The recoveries are compared to the method acceptance limits of 60-140%.

The MDL check sample method acceptance limits are 60-140%.

If the QC Reference check sample is outside of the method acceptance limits, a new initial calibration curve must be analyzed.

If any spike compound in the laboratory control sample falls outside of the acceptance limit window, the QC sample is considered to be out of control and any sample that is associated should be reextracted. However, if the recovery is high and the associated samples do not contain the specific compound(s), the data can possibly be accepted with narration.

If a spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable if matrix interference is indicated. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.

For DoD QSM 4.1, use QC acceptance criteria specified by DoD, if available. Otherwise use in-house control limits. In-house control limits must not be greater than $\pm$ 3 times the standard deviation of the mean LCS recovery. If the LCS fails the acceptance criteria, correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. If reanalysis cannot be performed, data must be qualified and explained in the narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.

For MS, when applying DoD QSM 4.1, apply J-flag to specific analyte(s) also in parent sample, if acceptance criteria not met. RPD must be $\leq$ 30% between MS and MSD.

8.4 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries are compared to laboratory established acceptance limits

When a sample has a surrogate that falls outside of the laboratory established acceptance limit window, the problem should be investigated. If the recovery looks like it is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reextracted.

For DoD QSM 4.1, use QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. When the surrogate recoveries fall outside of the acceptance criteria, apply Q-flag to all associated analytes.

8.5 DBCM: The check standard is run once per ICAL to indicate the retention time of DBCM which is close to that of the target analyte EDB.

8.6 NCR: Whenever data is not acceptable because of a failing LCS or surrogate recovery, a non-conformance report (NCR) must be initiated as soon as possible.

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) AND DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

Limits of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the anaysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of SW846 Method 8011 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste (SW-846), Third Edition, Method 8011 US EPA, Update III, December, 1996.

Methods for the Determination of Organic Compounds in Drinking Water, Supplement III, USEPA EMSL, EPA600/R-95-131, August 1995, Method 504.1.

Katahdin Analytical Services, Inc., SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) AND DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Current Version.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

LIST OF TABLES AND FIGURES

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) AND DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Six point initial calibration For DoD 4.1, minimum 5 point initial calibration	Prior to initial sample analysis and when continuing calibration standard can not meet criteria	Linear regression correlation coefficient $\geq$0.995	(1) Investigate source of problem (2)Recalibrate
QC Reference Sample	Once per week	60-140% Recovery	1) Investigate source of problem (2)Recalibrate
MDL Check Sample	Every Initial Calibration	60-140% Recovery	1) Investigate source of problem (2)Recalibrate
Method blank	One per prep batch of twenty or fewer samples	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are < PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS (check standard)	Equivalent to 10% of the sample load, or 1 per batch of samples extracted, whichever is greater	60-140% Recovery	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are < PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Calibration Verification Standard	Every 12-hour shift of operation, per 10 samples. At the beginning and/or end of analytical sequence.	70-130% Recovery	(1) Evaluate the samples: If the %R<70 or >130 and sample results are <PQL, narrate. If %R<70 or >130 only on one channel, narrate. If %R<70 or >130 and is likely a result of matrix interference, narrate. Otherwise, reanalyze all samples after the first failing CV.
Surrogate	Every field sample and QC sample	Refer to current GC Laboratory Established Acceptance Limit Sheet.	(1)Reextract sample if no detected surrogate recovery (2)Notate sample result if matrix interference indicated
Dibromochloromethane (DBCM)	Every Initial Calibration	Different retention time from EDB	Change instrument conditions in order to achieve resolution.

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) AND DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011

TABLE 1 (cont.)

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Matrix Spike/ Matrix Spike Duplicate	One for every set of 20 samples	60% -140 %Recovery and <30% RPD	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
Initial P & A study	Once per analyst	Method specified criteria	Repeat P & A study
MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) AND DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011

TABLE 2

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	Not Applicable (NA).	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from a 72-hour study.	NA.	NA.	
Breakdown check (Endrin / DDT Method 8081 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 15% for both DDT and Endrin.	Correct problem then repeat breakdown check.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 15% for both DDT and Endrin.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	One of the options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995; Option 3: non-linear regression: coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order). Mid point calibration of toxaphene and chlordane; if detected in sample, 6-point calibration is performed.	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin. Quantitation for multicomponent analytes such as chlordane, or toxaphene must be performed using a 5-point calibration. Results may not be quantitated using a single point.
Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) AND DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011

TABLE 2

DoD QSM REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification (ICV)	Immediately following ICAL.	All project analytes within established retention time windows. GC methods: All project analytes within ± 20% of expected value from the ICAL	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All project analytes within established retention time windows. GC methods: All project analytes within ± 20% of expected value from the ICAL	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported, including surrogates	One per preparatory batch.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements. In-house CLs may not be greater than ± 3 times the standard deviation of the mean LCS recovery. A number of analytes may fall outside the CL but within marginal exceedance limit depending on the total number of analytes in the LCS.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Refer to Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) AND DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix if sufficient sample is available.	For matrix evaluation, use laboratory LCS CLs or use DoD-generated LCS-CLs, if available depending on project requirements.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix Spike duplicate (MSD)	One per preparatory batch per matrix if sufficient sample is available.	MSD: For matrix evaluation, use laboratory LCS CLs or use DoD-generated LCS-CLs, if available depending on project requirements. MS/MSD: RPD ≤ 30%.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	The laboratory shall use laboratory surrogate CLs or use DoD-generated surrogate CLs, if available depending on project requirements.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepped within hold time.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method reporting requirements; otherwise, report the result from the primary column (see Box D-16).
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) AND DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-319-09	METHOD 8011, current revision
Procedure	1) 7.4.2 Remove the container cap and add 6 g of NaCl to all the samples. 2) Katahdin does not perform this step.	1) 7.4.1 Remove the container cap and add 7 g of NaCl to all the samples. 2) 7.4.5 Transfer the remaining hexane phase, being careful not to include any of the water phase, into a second vial. Reserve this second vial at 4^{o}C for reanalysis if necessary.
QC-Frequency of check standard, DBCM and MDL Check Sample	8.1 … For *every initial calibration* curve that is prepared and analyzed, a *DBCM* and a *MDL check sample* are analyzed….	*There is no reference to a MDL check sample or a DBCM.* *The check standard or LCS is* 8.3 The laboratory must demonstrate on a frequency to 5% of the sample load or once per analytical batch…
QC-Surrogate	Surrogate Tetrachloro-m-xylene is added to all samples, blanks, and standards.	No reference to a surrogate.
Target compound identification	7.5.4 …If an analyte is present but its retention time is ±0.04 minutes or more than the retention time of the analyte in the preceding CV, then the analyte is undetected electronically in Target.	7.7.1 Identify EDB and DBCP in the sample chromatogram by comparing the retention time of the suspect peak to retention times generated by the calibration standards and the check standard.

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) AND DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011

Table 4

PRATICAL QUANTITATION Limits

Analyte	(ug/L)
1,2-Dibromoethane	0.05
1,2-Dibromo-3-Chloropropane	0.05

- In some case (ie. South Carolina NPDES work) a lower PQL may be required. In these cases a calibration point at 0.01 ug/L shall be analyzed and the PQL can be changed to 0.02ug/L

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) AND DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011

FIGURE 1

PREPARATION LOGBOOK

KATAHDIN ANALYTICAL SERVICES

Method (circle) EPA 504.1 SW846 8011
Matrix: Aqueous
Spike ID's: CV/ICAL/LCS STD A: P6849
CV/ICAL/LCS STD B: P6850
QC STD C: P6852
SURR STD: P6861
DBCM STD: P6704
Hexane Lot #: 06377
NaCl Lot #: 15C289
Balance ID: Mettler PJ400
Start time: 1160 End Time: 1150

Date Ext.	Init.	Sample ID	Samp + Vial g.	Vial g.	Sample Amt. mL.	Surr. Vol. uL.	Spk Vol. uL.	Final Vol. mL.	Comments
3-14-13	AC	ICAL 0.05	NR	NR	35	25	25 D	2	R227156
		0.1				37.5	50		
		0.25				50	125		
		0.5				62.5	250		
		1.0				125	50 A		
		2.5				250	125		
		QC IND				125	125 C		
		MDL					25 B		
		DBCM					500		
		WG121533-1					NR		
		-2					125 B		
		-3							
		SG1626-1E	64.20	30.36	33.84		NR		
		-3A	60.10	25.50	34.60				
							AC 3-20-13		

GC-011 - Revision 1 - 06/23/2010 QAGC272 000002

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) AND DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011

FIGURE 2

INSTRUMENT RUN LOG

Katahdin Analytical Services, Inc.

GC Laboratory Instrument Runlog
Instrument: GC01
Amount Injected: 2 uL
Column Numbers: 347/348
Method: SW846 (8081) / 8082 / 8151
(circle) EPA (504.1) / 556 / 608

Standard	Standard ID
EVAL	P6737
INDAB 0.05	P6830
↓ 0.025	P6831
TDX 1.0	P6854

Date	Init.	Result File	Sample ID	Y/N	Analytical Workgroup	Method	Comments
3-8-13	JLP	1GC00030	WG-121029-1 3544	Y	WG-121130	Pest102	
		31	-2				2↓ATB
		32	-3				2↓ATB
		33	-4				
		34	SG1392-2 ↓	↓			ICAL both
		35	INDAB 0.025	Y	-7,8		
		36	TDX 1.0		-9,10		
		37	WG-121136-1 3510				
		38	-2				
		39	-3				
		40	-4				
		41	SG1392-9RE ↓				~~SDA both~~ JLP 3/11/13
		42	INDAB 0.05		-11,12		ATA5TB
↓	↓	↓ 43	TDX 1.0	↓	↓ -13,14	↓	
3/11/13	CB	1GC00044	RINSE	N		504070	
		45	ICAL 0.05	Y	WG121224-4,5		
		46	0.1		-6,7		
		47	0.25		-8,9		
		48	0.5		-10,11		
		49	1.0		-12,13		
		50	↓ 2.5	↓	-16,17		
		51	QC IND	Y			
		52	MDL				
		53	DBCM	↓			
		54	WG121224-1	Y			
		55	-2				
		56	↓ -3				
		57	SG1500-1	↓			DCB A
↓	↓	↓ 58	CV 0.5	Y	↓ -14,15	↓	
3-19-13	AC	↓ 59				504071	

GC-004 – Revision 2 – 05/15/2012

QAGC263

0000086

TITLE: EXTRACTION AND ANALYSIS OF EDB (1,2-Dibromoethane) AND DBCP (1,2-Dibromo-3-Chloropropane) IN WATER ACCORDING TO SW846 METHOD 8011

FIGURE 3

DATA REVIEW CHECKLIST

PRIMARY REVIEW CHECKLIST

Verbal Due Date _____ (☐ Verbals turned in). Due Date_____

Client:		Primary	Secondary
Method:		Date:	Date:
SDG No:	Level:	Initials:	Initials:
KAS No:			Approved : ☐ Yes

DODQSM (4.1) ☐ DOD W/ LAB. LIMITS ☐ QUAPP ☐ LAB ☐
(*REPORT ND's to - POL* ☐ *MDL* ☐ *LOD* ☐)

List all curves that ***are scanned*** (hard copy not included). _____

_____ _____ _____ _____

Narrate which QC limits were used for (Surr., LCS's MS/MSD's.)	☐
All needed forms are present .	☐
Correct Work Order Number or SDG name (all forms).	☐
Correct project name and spelling (all forms). (Truncated ☐).	☐
Correct file numbers (all forms).	☐
Analysis Date Correct.	☐
Extraction Method & Analysis Method Correct.	☐
Product list compared to ROAs (compounds & PQLs).	☐
Chromatogram reviewed for unlabeled peaks (check product list).	☐
Flagging of all ROAs correct (Florida ☐) (Florida ☐).	☐
All tunes included (level IV) .	☐
All log book pages included (Soil weights,TCLP & SPLP).	☐
Verify DOD QSM criteria and/or Project specific requirements.	☐
Narrate any method deviations. (Blanks, LCS's etc.)	☐
Sign & Date Manual integration **(Narrate as needed).**	☐

Sample I.D's Truncated (**NARRATED**). YES ☐ Please list KAS # below :

_____

QA-044 – Revision 3 – 10/24/2012

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS or DIESEL RANGE ORGANICS (DRO)

Prepared By: Mike Thomas Date: 6/97

Approved By:

Group Supervisor: Michael F. Thomas Date: 1/29/01

Operations Manager: Joh C. Benton Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1.29.01

General Manager: Dennis P. Hoffman Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Format changes, added pollution prevention, removed Soil extraction, other minor changes to sections 7, 8 and QA Table.	DN	1.29.01	1/29/01
02	Wording added or changed to clarify sections 5, 6, 8, + 9. Minor changes throughout. New figure.	MRC	11.08.04 ~~11.08.04~~ MRC	11.08.04
03	Added - the condenser temperature during extraction. Added - variable transformers output should be set at 55%. Updated Logbook	LAD	04/06	04/06
04	Added waste generated information. Added Acetone to reagents. Added QC pH must be adjusted. Added use of boiling stones in Sox. ext. Removed NaCl2 references. Updated Table 2. Added definitions	LAD	09/07	09/07
05	updated Logbook, added recording of lot numbers and nitrogen evaporation water bath temperature in logbook	LAP	11/08	11/08

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS or DIESEL RANGE ORGANICS (DRO)

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added commercially available DRO product to section 5. Updated logbook page.	LAD	08/10	08/10
07	Minor changes to Section 7 to reflect current practices. Removed fluted from filter paper type. Updated MDL, LOD, LOQ information. Added DoD and NELAC References. Removed Sect. 8.4 and added it to Method Modifications.	LAD	04/12	04/12

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS or DIESEL RANGE ORGANICS (DRO)

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-520-07**, titled **Preparation of Aqueous Samples for Analysis of Extractable Petroleum Hydrocarbons or Diesel Range Organics (DRO).**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-520-07**, titled **Preparation of Aqueous Samples for Analysis of Extractable Petroleum Hydrocarbons or Diesel Range Organics (DRO).**

Recipient: _____ Date: _____

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS or DIESEL RANGE ORGANICS (DRO)

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the method used by Katahdin Analytical Services technical personnel to prepare samples for analyses of Total Petroleum Hydrocarbons (TPH) or Diesel Range Organics (DRO) in water. These compounds correspond to a hydrocarbon range of C9-C36 inclusive and a boiling point range between approximately 170°C and 430°C. The method is based on a solvent extraction procedure followed by Gas Chromatography (GC) analysis.

The method is designed to measure “mid-range” to “heavy” petroleum products. This range would include JP-4, JP-5, JP-8, kerosene, diesel #2, #4, #6, and motor oil. Components greater than C36 present in products are not readily amenable to this method. If, based on a review of the chromatogram, the presence of these product types is suspected, a qualitative description should be included in the report. Additional analyses may be performed including, but not limited to, analysis of additional reference materials. These additional efforts are not contained within this method.

1.1 Definitions

TOTAL PETROLEUM HYDROCARBONS (TPH): All resolved and unresolved material eluting from n-nonane (n-C9) through n-hexatriacontane (n-C36), inclusive.

DIESEL RANGE ORGANICS (DRO): All resolved and unresolved material eluting from decane (n-C10) through n-octacosane (n-C28), inclusive.

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative

percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of water samples for the determination of petroleum hydrocarbons or DROs. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability," current revision, and Section 8.2.

It is the responsibility of all Katahdin technical personnel involved in the extraction of water and soil samples for the determination of petroleum hydrocarbons or DROs to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall

receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone is considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Sodium sulfate waste should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

Samples are extracted with methylene chloride using separatory funnels following EPA Method 3510 or continuous liquid liquid extractors (CLLE) 3520 for subsequent analysis for Total Petroleum Hydrocarbons or Diesel Range Organics. The extract is dried, concentrated and injected into a capillary column gas chromatograph. This method is suitable for the analysis of aqueous samples.

This method is based in part on: 1) Petroleum Hydrocarbon Methods by API revised August 1993; 2) The Wisconsin DRO method, 3) SW-846 methods 3510, 3520, 3540, and 3550, 4) The Massachusetts EPH method; and 5) The Maine DRO method.

3.0 INTERFERENCES

3.1 Other organic compounds including chlorinated hydrocarbons, phenols, and phthalate esters are measurable by this method. As defined in the method, the DRO results include these compounds. Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interferences, further cleanup of the sample extract may be needed to minimize interferences.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS or DIESEL RANGE ORGANICS (DRO)

3.2 Method interferences are reduced by washing all glassware with hot soapy water and then rinsing it sequentially with tap water, methanol or acetone, and methylene chloride. Method/reagent blanks (Surrogate Control Samples) must be analyzed with each batch to demonstrate that the samples are free from method interferences.

3.3 High purity reagents such as Burdick and Jackson GC methylene chloride or Baker capillary grade methylene chloride must be used to minimize interferences.

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with the solvent to be used for extraction.

4.1 Separatory Funnel - 2000 mL capacity, Nalgene Teflon FEP separatory funnels with Nalgene Tefzel® screw-cap closures (or equivalent)

4.2 Concentrator tube - 10 mL, graduated

4.3 Evaporative flask - Kuderna-Danish, 500 mL capacity attached to concentrator with neck clips

4.4 Snyder column - Kuderna-Danish, three ball macro

4.5 Graduated cylinders - 100 mL, 1000 mL, or 2000 mL

4.6 Short stem funnels

4.7 250 mL amber collection bottles with Teflon-lined caps

4.8 1.8 mL, 12 mL and/or 16 mL glass vials with Teflon-lined caps

4.9 Continuous liquid-liquid extractors (CLLE) including body, 500 mL flat bottom boiling flask and Alhin condensers

4.10 Filter paper, 18.5 cm, Fisher brand or Whatman #4 (or equivalent)

4.11 Nitrogen evaporation apparatus.

4.12 Boiling chips - approximately 10/40 mesh, Teflon or selenized carborundum, 12 mesh (or equivalent).

4.13 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer.

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5.0 REAGENTS

5.1 Reagent water - water in which an interferent is not observed at or above the PQL for any parameter of interest (laboratory reagent grade water or equivalent).

5.2 Sodium sulfate (granular, anhydrous and powdered, anhydrous) (ACS reagent grade), Na_2SO_4 . Certified by the manufacturer/vendor as purified by heating at 400°C for 4 hours prior to receipt by the laboratory.

5.3 Acid for preserving water samples: A 1:1 mixture of reagent water and concentrated hydrochloric acid. Use ~5 mL per 1 L sample.

5.4 Methylene Chloride ($MeCL_2$) and Acetone - Pesticide grade or better. Lots must be verified by concentrating 300-400 mL to 1.0 mL and evaluating by GC/MS.

5.5 Surrogate spiking solution - Prepare a solution of o-Terphenyl at a concentration of 20 ug/mL in acetone. Store the solution at -10 to -20 °C in a Teflon sealed container. Solution must be verified by GC/FID prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.6 Matrix Spike/Lab Control Sample spiking solution - Prepare a matrix spiking solution in pesticide grade Acetone that contains all target analytes listed below:

Component	Concentration µg/mL
Decane	50
Dodecane	50
Tetradecane	50
Hexadecane	50
Octadecane	50
Eicosane	50
Docosane	50
Tetracosane	50
Hexacosane	50
Octacosane	50

Alternatively, a Matrix Spike/Lab Control Sample spiking solution may be prepared using a commercially available fuel oil such as Diesel Fuel #2, unweathered, from Restek. Prepare a 500 ug/L standard in pesticide grade Acetone. This spike may be required for by some certain states, federal programs, or clients (such as South Carolina).

Store the both solutions at –10 to -20 °C in a Teflon sealed container. The solutions must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

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6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Aqueous samples are collected in a 1L amber glass bottle. As soon as possible after samples are received they must be stored at 4°C (±2°C) until extraction. When water samples are not received cold, the fact should be noted on the chain of custody form. The pH of aqueous samples must be checked with pH paper upon receipt to ensure that the samples have been acid preserved. If the pH is not < 2, the fact should be noted on the chain of custody form. Samples that have not been preserved should be preserved at this time and a notation made on the chain of custody form.

Holding time for extraction of aqueous samples for Methods 3510 and 3520 is 7 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook (all that are applicable).

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- pH
- Extraction and concentration dates
- Extraction and concentration analyst
- Separatory funnel extraction start and end times.
- CLLE extraction start and end times, also the prep start and end times.
- Sample ID or QC sample ID
- Initial and final volumes
- Surrogate and spike amounts
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

SEPARATORY FUNNEL SAMPLE EXTRACTION

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interference, then samples should be extracted by continuous liquid-liquid extraction (CLLE).

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7.1 Rinse all glassware three times with methylene chloride prior to use.

7.2 Label a 2 L Teflon separatory funnel and a 250 mL amber collection bottle clearly. Label should include laboratory sample number, matrix, analyte, and extraction date. Be sure that the detachable stopcocks are secured to the separatory funnels before adding samples.

7.3 Measure the initial volume by comparing the meniscus of the sample with the reference bottle of the same bottle type. Please refer to SOP CA-108, "Basic Laboratory Technique", for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.4 Transfer the contents of the sample bottle to a 2 L separatory funnel.

7.5 Transfer 1 L of reagent water to a 2 L separatory funnel. This serves as a method blank for the extraction batch. A method blank must be prepared for every daily extraction batch of twenty or fewer samples.

7.6 Transfer 1 L of reagent water to a 2 L separatory funnel. This will serve as a Laboratory Control Sample (LCS). An LCS is required for every daily extraction batch of twenty or fewer samples. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.7 A matrix spike/matrix spike duplicate (MS/MSD) is to be prepared as requested by a client or, at a minimum, one pair per 20 samples if there is sufficient sample volume. Measure the initial volume as in 7.3. Transfer two additional 1 L aliquots of sample to 2 L separatory funnels for a matrix spike and matrix spike duplicate (MS/MSD).

Note: Sufficient sample volume should be available without depleting all remaining sample aliquots.

7.8 Check to make sure the pH is <2. Note in the prep logbook if the pH is not <2 and adjust if needed with 1:1 HCl. (This should have been recorded and corrected, if necessary, at the time of sample receipt by the sample custodians.)

7.9 Adjust method blank and LCS/LCSD pH to <2 with 1:1 HCl.

7.10 Using a gas-tight syringe, add 1.0 mL of surrogate spiking solution to all samples the blank, LCS/LCSD and MS/MSD, if performed.

7.11 Using a gas-tight syringe, add 1.0 mL of matrix spiking solution to each LCS/LCSD and MS/MSD.

7.12 Carefully add 60 mLs of methylene chloride to each empty sample bottle rinse the bottle and transfer the solvent into the appropriate separatory funnel making sure the dispenser tip does not come in contact with the bottle. Add 60 mL of methylene chloride directly to the blank and LCS/LCSD.

7.13 Ensure that each screw-cap is secured tightly to the separatory funnel to prevent leaks. Extract the sample by first shaking the funnel by hand for a few seconds, venting often in a hood to release pressure. Place funnel on mechanical shaker and shake for 3 minutes. Allow phases to separate. Drain the methylene chloride layer into the 250 mL amber collection bottle.

7.14 If an emulsion forms, mechanical techniques must be employed to achieve maximum separation and solvent recovery. Such means include swirling and centrifugation and draining through a small separatory funnel. In certain instances, transferring the entire sample into a continuous liquid-liquid extractor may be the only alternative. If any such techniques are used, they must be noted in the extractions logbook.

7.15 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time (see 7.11 - 7.12). Collect the methylene chloride layer in the same 250 mL amber collection bottle.

7.16 Repeat the extraction for a third time as described in 7.13.

7.17 Proceed to Section 7.32 for extract concentration procedures.

CONTINUOUS LIQUID-LIQUID SAMPLE EXTRACTION (CLLE)

7.18 Set up the CLLE apparatus and add one or two boiling stones to the flask. All glassware should be rinsed three times with methylene chloride and the extract flasks properly labeled.

7.19 Add approximately 500 - 600 mL of methylene chloride to the CLLE body.

7.20 Add 1 L reagent water to a CLLE body. This is the method blank for this extraction batch. Be sure that no water leaks into the round bottom flask. A method blank is required for every extraction batch of twenty or fewer samples.

7.21 Prepare an LCS for every daily extraction batch of twenty or fewer samples. Add 1 L of reagent water to a CLLE body. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

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7.22 Check to make sure the pH is <2. Note in the prep logbook if the pH is not <2 and adjust if needed with 1:1 HCl. (This should have been recorded and corrected, if necessary, at the time of sample receipt by the sample custodians.)

7.23 Determine the initial volume as in 7.3. Transfer the samples to the CLLE bodies, being sure no sample leaks into the round bottom flask.

7.24 Transfer two 1 L portions of a sample to CLLE bodies for preparation of a matrix spike/matrix spike duplicate if required. An MS/MSD is required if requested by the client or per 20 samples or every 14 days, whichever occurs first. (Refer to the logbook page, "date QC expires"). Note: Sufficient sample volume should be available without depleting all remaining sample aliquots.

7.25 For each sample, rinse the original sample container with approximately 30 mL of methylene chloride being careful not to touch the bottle with the dispenser tip. Add this rinse to the CLLE body.

7.26 Adjust method blank and LCS/LCSD pH to <2 with 1:1 HCl.

7.27 Add 1.0 mL of the surrogate spiking solution to each sample including the blank, LCS/LCSD and MS/MSD, if performed.

7.29 Add 1.0 mL of matrix spiking solution to the appropriate LCS/LCSD and MS/MSD pair, if performed, and stir.

7.30 Rinse each 45/50 condenser joint with methylene chloride. Attach cooloing water Alhin condensers set to a temperature of 15 °C. Turn on the heating mantles, the rheostat of the variable transformers should be set to 55% of the output voltage. Allow the samples to extract for 20 $\pm$ 2 hours. Turn off the mantles and let samples cool.

7.31 Proceed to Section 7.32 for sample extract concentration procedures.

CONCENTRATION OF WATER SAMPLE EXTRACTS

7.32 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add a few boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Place the assembled K-D's under the funnels.

7.33 Transfer the methylene chloride extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract

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volume through the sodium sulfate, rinse the extract bottle three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow to drain.

7.34 Transfer the labels from the collection bottles or round bottom flasks (from the CLLE extraction) to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.35 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 5-6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.36 Reduce the methylene chloride extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging needle closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.

7.37 Reduce each extract to slightly less than 1 mL and then, using a 5 ¾" pasteur pipet, transfer the final extract to a properly labeled 1.8 mL vial with PTFE-lined cap. Adjust the final volume of each extract to 1 mL using the 1 mL oil-filled reference vial for volume comparison. If the extract is highly colored or a precipitate forms during concentration, the final volume should be adjusted and noted in the extractions logbook with a comment.

7.38 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the "tray location" of the individual extract vials.

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8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

8.1 If a solvent blank or extraction blank is above the reporting level all associated samples with the "dirty" blank must be carefully evaluated versus the blank contamination level. Samples that contain DRO at a level of ten times or more than the blank level may be useable. If the sample level is less than ten times the blank level the source of the contamination is not as certain for the samples and they should be re-extracted after consultation with the client.

8.2 The analyst must make an initial demonstration of the ability to generate acceptable accuracy and precision with this method by successful analysis of the following:

8.2.1 Replicate commercial diesel oil spikes in water: Analysis of at least 4 replicates at a concentration of 100 µg/L (in water) with accuracy of the replicates falling between 60% to 140% of the known concentration. The precision of all replicates should be within 20%.

8.3 ME-DRO method LCS Requirements: every 20 water samples analyzed, the lab must analyze a set of duplicate diesel component spikes in reagent water. The Duplicate spikes must be run through the method in the same manner as samples. The accuracy of the two water spikes should fall between 60% to 140% of the known concentration with a relative % difference of 20% or less. **Alternatively duplicate samples and spiked samples can be substituted for the laboratory spiked duplicates at a frequency of 10%. Care must be taken to ensure that the samples are homogeneous before analyzing duplicates and spikes.**

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency

must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Detection (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all

parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Detection limit for waters: The laboratory must be able to achieve a detection limit of 50 µg/L using a commercial diesel fuel oil mixture spiked into laboratory blank water and calculated against the DRO component standard.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

ASTM "Std Mtds for Comparison of Waterborne Petroleum Oils by GC," 3328-78.
Wisconsin DNR Modified DRO method, July 1993, Revision 6.

USEPA SW 846, 3rd edition, Methods 8000, 8100, 3500, 3510, 3520 and 3550.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Current Version.

LIST OF TABLES AND FIGURES

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS or DIESEL RANGE ORGANICS (DRO)

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Sample Prep for waters and soils for DRO or TPH determination	Method blank	One per prep batch or 20 samples whichever is more frequent.	Refer to analytical method.	Refer to analytical method.
	LCS	One per prep batch	Refer to analytical method.	Refer to analytical method.
	Routinely Matrix Spike/Matrix Spike Duplicate	One set for every 20 samples or 14 days whichever is more frequent, given sufficient sample volume.	Refer to analytical method.	Refer to analytical method.
	Per client Request Sample Duplicate/ Matrix Spike	One sample duplicate per twenty samples in conjunction with a matrix spike per 20 samples or 14 days whichever is more frequent.	Refer to analytical method.	Refer to analytical method.
	Demonstration of analyst proficiency; accuracy and precision	One time per analyst initially, and annually thereafter	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file
	MDL, LOD and LOQ studies and verifications	Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit and Instrument Detection Limit Studies, for procedures on determining the MDL, LOD and LOQ.		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR ANALYSIS OF EXTRACTABLE PETROLEUM HYDROCARBONS or DIESEL RANGE ORGANICS (DRO)

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-520-07	METHOD
Apparatus/Materials	1. 250 mL amber bottle used for extract collection 2. 1 mL syringe 3. Short stem funnels	1. 250 mL Erlenmeyer flasks 2. 5 mL syringe 3. Drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. Extract collection in amber bottle or Erlenmeyer flask 2. Add surrogate/spike to sample in CLLE 3. Extract for 3 minutes on mechanical shaker 4. Extract dried using Na_2SO_4 in short stem funnels 5. Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 6. Water bath temp 75-85 deg C 7. No apparatus height specification for concentration on water bath 8. Sample removed from water bath when volume reaches ~6 mL 9. N bath temp no higher than 39 deg C	1. Extract collection in Erlenmeyer flask 2. Add surrogate/spike directly to sample bottle 3. Extract by shaking vigorously for 1 - 2 minutes with periodic venting 4. Extract dried using Na_2SO_4 in drying columns 5. Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer 6. Water bath temp 15-20 deg C above solvent boiling temp 7. Partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min 8. Sample removed from water bath when volume reaches 1 mL 9. N bath temp 35 deg C
QC - Spikes		
QC - LCS	KAS does not perform.	Laboratory spiked duplicates prepared by spiking fuel oil into blank water in must be run at a minimum frequency of 5%. Alternatively duplicate samples and spiked samples can be substituted for the laboratory spiked duplicates at a frequency of 10%. Care must be taken to ensure that the samples are homogeneous before analyzing duplicates and spikes.
QC - Accuracy/Precision		
QC – MDL		

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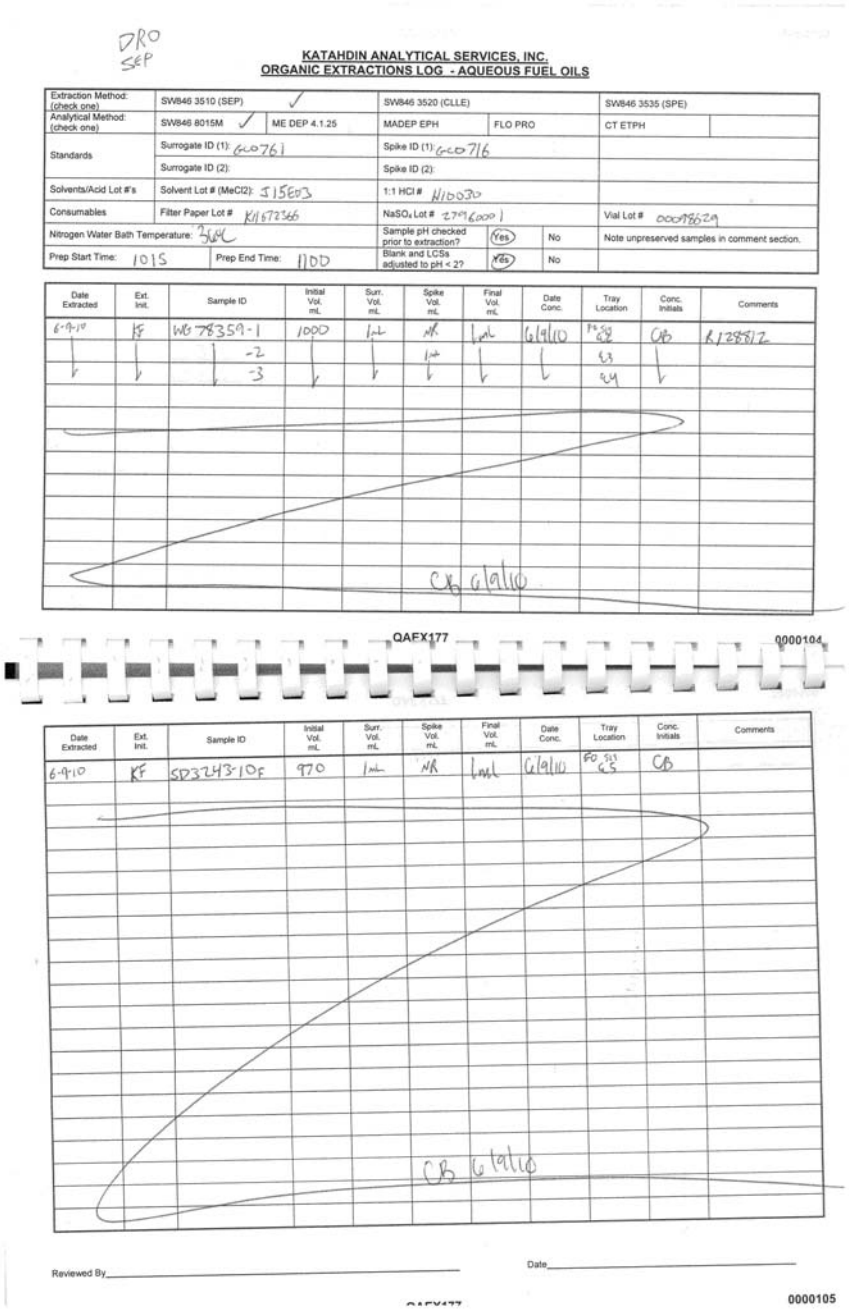
FIGURE 1

EXAMPLE OF EXTRACTION LOGBOOK PAGE



DRO SEP

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - AQUEOUS FUEL OILS

Extraction Method: (check one)	SW846 3510 (SEP) ✓		SW846 3520 (CLLE)		SW846 3535 (SPE)		
Analytical Method: (check one)	SW846 8015M ✓	ME DEP 4.1.25	MADEP EPH	FLO PRO	CT ETPH		
Standards	Surrogate ID (1): 60761		Spike ID (1): 60716				
	Surrogate ID (2):		Spike ID (2):				
Solvents/Acid Lot #'s	Solvent Lot # (MeCl2): J15E03		1:1 HCl # H10030				
Consumables	Filter Paper Lot # K1672366		$NaSO_4$ Lot # 2796000 1		Vial Lot # 00078629		
Nitrogen Water Bath Temperature: 36°C			Sample pH checked prior to extraction?	Yes / No	Note unpreserved samples in comment section.		
Prep Start Time: 1015	Prep End Time: 1100		Blank and LCSs adjusted to pH < 2?	Yes / No			

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol. mL	Spike Vol. mL	Final Vol. mL	Date Conc.	Tray Location	Conc. Initials	Comments
6-9-10	KF	WG78359-1	1000	1mL	NA	1mL	6/9/10	FO 5B 62	CB	R128812
"	"	-2	"	"	1mL	"	"	63	"	
"	"	-3	"	"	"	"	"	64	"	

CB 6/9/10

QAFX177 0000104

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol. mL	Spike Vol. mL	Final Vol. mL	Date Conc.	Tray Location	Conc. Initials	Comments
6-9-10	KF	SD32413-10F	970	1mL	NA	1mL	6/9/10	FO 5B 65	CB	

CB 6/9/10

Reviewed By_____ Date_____

0000105

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Jessica Spearhawk-Wildes

Review Date: 3-15-13

SOP Number: GA-520

SOP Title: Preparation of aqueous samples for analysis of extractable petroleum hydrocarbons or DRO

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 3-15-13

QAO Signature: Jessie Dimond **Date:** 032113

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-333
Revision History
Cover Page
Page 1

TITLE: DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO

Prepared By: Peter Lemay Date: 7/18/01

Approved By:

Group Supervisor: Peter Lemay Date: 7/18/01

Operations Manager: John Burley by Deborah Nadeau Date: 7/18/01

QA Officer: Deborah J. Nadeau Date: 7.18.01

General Manager: Deborah F. Nadeau Date: 7/18/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Added definitions and information for new data processing system. Added or changed wording to clarify sections 6 and 7 and Table 2. Added wording to sections 8 and 9 per recent NELAC + Navy audits. Minor changes throughout. New figures 1 and 2.	MRC	11.15.04	11.15.04
02	Changed LIMS to KIMS Sodium Sulfate is purified at vendor added wording to sect. 7.7.2 to clarify	LAD	03/06	03/06
03	Many changes made throughout, including but not limited to, waste management, CV frequency, Spike amounts, statistically derived QC limits and method modifications. Please refer to the QAM/SOP change form filed with the SOP in QA for a detailed list of changes	LAD	09/07	09/07
04	Removed Apparatus and Reagents that are not used. Updated surrogate information	LAD	09/08	09/08
05	Sect. 8.4 and Table 1 - Changed statistical limits to method limits. Added references. Updated Data Review Checklist	LAD	09/10	09/10

TITLE: DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	8.6 – Changed CAR to nonconformance report. Section 9 - Added MDL/LOD/LOQ information. Updated Runlog and Review Checklist examples.	LAD	01/12	01/12
07	Sect. 1 & 7 - changed reporting from Quick-forms to KIMS. Updated Fig. 2 - Review Checklist. Minor edits.	LAD	05/13	05/13

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ____ of document **SOP CA-333-07**, titled **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-333-07**, titled **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

Recipient: _____ Date: _____

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the method used by Katahdin Analytical Services technical personnel to measure the concentration of petroleum range organics (PRO) in water and soil. These compounds correspond to a hydrocarbon range of C_8-C_{40}.

This method is based on a solvent extraction, Gas Chromatography (GC) procedure. The method is designed to measure the petroleum concentration in environmental samples in the above stated C-Range (nominally diesel through motor oils). It cannot be used as an indication of gasoline contamination. Additional analyses may be performed including, but not limited to, analysis of additional reference materials. These additional efforts are not contained within this method.

1.1 Definitions

PETROLEUM HYDROCARBONS: All chromatographic peaks, both resolved and unresolved, eluting between the peak start of n-octane (n-C_8) and the peak end after n-tetracontane (n-C_{40}). Quantitation is based on direct comparison of the area within this range to the total area of the Petroleum Hydrocarbon standard as determined from FID response using baseline-baseline integration.

PETROLEUM HYDROCARBON STANDARD: A 17-component mix of all even numbered normal alkanes from C8 to C40. This standard serves as a quantitation standard and a retention time window defining Petroleum Hydrocarbons.

SAMPLE MATRIX SPIKE: A selected sample from the analytical batch spiked with the Petroleum Hydrocarbon Standard and surrogate standards. The calculated spike recovery shall be used as a control.

LABORATORY CONTROL SAMPLE: Laboratory reagent grade water or standard soil spiked with the Petroleum Hydrocarbon standard and surrogate standards. The calculated spike recovery may be used as a laboratory control.

METHOD DETECTION LIMIT (MDL): Minimum concentration that an analyte can be measured and reported with a 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. The MDL is determined using EPA Appendix B to Part 136, CFR 40 Ch. 1(7-1-94) using the Student t Test.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. LIMS utilizes these features through a database.

PE NELSON TURBOCHROM: A data acquisition system that is used to collect chromatographic data. The system can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of PRO by Method FL-PRO. Analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of PRO by Method FL-PRO to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual, including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and

lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the preparation of standards etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. FLPRO sample vials are considered "P" waste and should be disposed of in the corresponding satellite waste accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 One liter of water or a specified quantity of soil (extraction method dependent) is spiked with two surrogates and extracted with Methylene chloride. The water is removed from the extract, concentrated to a volume of 2.0 mL, and treated with silica gel to remove potential organic interferences. An aliquot is injected onto a capillary column gas chromatograph (GC) equipped with a flame ionization detector (FID). Quanitation is based on the detector response compared to a series of normal alkane standards. This method is suitable for the analysis of waters, soils or wastes.

2.2 This method is based in part on USEPA Methods 8000 and 8100, SW-846, "Test Methods for Evaluating Solid Waste", 3rd Edition, Method OA-2, work by the EPA UST Work Group "Measurement of Petroleum Hydrocarbons: Report on activities to Develop a Manual", 1990, Method AK103.0, Revision 2, PUBL-SW-141, July 1993

and the Florida Department of Environmental Protection Technical Advisory Committee for 62-770, F. A. C, Petroleum Contamination Site Cleanup Criteria.

3.0 INTERFERENCES

3.1 Other organic compounds including chlorinated hydrocarbons, phenols, and phthalate esters are measurable. As defined in the method, the PRO results include these compounds. Spills of known specific constituents should be analyzed and quantified by a method specific for those compounds.

3.2 Method interferences are reduced by washing all glassware with hot soapy water and then rinsing it sequentially with tap water, methanol or acetone, and Methylene chloride. Method blanks must be analyzed with each batch to demonstrate that the samples are free from method interferences.

3.3 High purity reagents (pesticide grade or better) must be used to minimize interferences.

3.4 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it should be followed by analysis of a solvent blank to check for cross-contamination.

3.5 Animal and vegetable oil and grease and biogenic terpenes are also measurable if the sample is not cleaned up before analysis. In order to eliminate false positives from these sources, the silica cleanup is a mandatory part of the procedure.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph: Analytical system complete with gas chromatograph and all required accessories, including a detector, column supplies, recorder, gases, and syringes. A capillary split/splitless injector operating in the splitless mode is recommended. A data system capable of determining peak areas by integrating from baseline to baseline is required.

4.2 Column 1: 30 m x 0.53 mm ID ZB-5, 1.5 micron film thickness (or equivalent). Column 2: 30 m x 0.53 mm ID ZB-1, 1.5 micron film thickness (or equivalent). The column must be capable of resolving typical diesel components, and the solvent front from C_8. Other columns may be used if all column performance criteria are met.

4.3 Detector: Flame ionization detector (FID).

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

4.4 Microsyringes: 1 ul, 5 ul, 10 ul, 25 ul, and 100 ul.

4.5 Disposable pipettes: Pasteur.

4.6 2 ml (and larger) vials with Teflon lined caps for storage of extracts.

5.0 REAGENTS

5.1 Solvents: Methylene chloride: Pesticide grade or equivalent. Store away from other solvents.

5.2 Stock Standards: Aliphatic Hydrocarbon standard mix from a vendor like UltraScientific at a concentration of 500 ug/mL in hexane (each of the 17 components from C_8 to C_{40}). A surrogate solution containing n-Triacontane-d_{62} at a concentration of 5000 ug/mL and another surrogate solution containing o-Terphenyl at a concentration of 2000 ug/mL from a vendor like Restek.

5.3 Calibration Standards: The standards are prepared at the following five different concentrations: 200 ug/ml, 100 ug/ml, 50 ug/ml, 20 ug/ml, and 5 ug/ml (per each component). This is equivalent to 85, 340, 850, 1700, and 3400 ug/ml total alkanes in the standards. The concentration of OTP and triacontane-d_{62} must remain at a constant 50 and 300 ug/ml level in all concentration levels.

5.3.1 Transfer the stock standard solution into a Teflon-sealed screw-cap/crimp cap bottle. Store, with minimal headspace, at 6°C or less and protect from light.

5.3.2 Working standards must be replaced after 6 months, or sooner if comparison with check standards indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Whenever possible, samples should be grab samples which are collected directly into the sample container. Sample collection equipment such as bailer or intermediate containers should be avoided (exceptions: collection from monitoring wells or grab samples in surface water at depth). Unless required by permit, automatic samplers may not be used. Pumps such as bladder pumps or peristaltic pumps shall not be used.

6.2 All sampling equipment which contacts the sample shall be constructed of teflon®, stainless steel or glass. Under no circumstances can flexible PVC tubing, such as tygon®, be used in the purging or sample collection process.

6.3 Water samples shall be collected in a one liter glass container; soils in a glass jar. All containers shall be sealed with a screw cap with teflon® liner. Water samples shall be acidified to a pH of less than 2 with hydrochloric or sulfuric acid (reagent grade or better).

6.4 The samples shall be stored at 4°C (±2°C) from the time of collection until extraction. Extraction shall be performed on waters within seven days of sample collection and on soils within 14 days of sample collection. All analyses must take place within 40 days of extraction.

7.0 PROCEDURES

7.1 Waters are extracted using a separatory funnel or continuous liquid liquid extraction technique. Soils are extracted using a sonication technique. Alternatively, soils may be extracted by a Soxhlet extraction technique. Refer to Katahdin SOP CA-520, current revision, for sample preparation procedures. After the extracts are concentrated, an appropriate volume (usually 1ul) is injected directly into the GC. (Recommend using splitless injection techniques).

NOTE: NaCl may be added to water samples to improve extraction efficiency.

If the sample concentration exceeds the calibration range for PRO an appropriate dilution should be used. An appropriate dilution is one that keeps the response of major constituents (previously saturated peaks) in the linear range of the detector. If an initial dilution does not accomplish this then an intermediate dilution should be performed.

7.2 Gas Chromatography:

7.2.1 Conditions (For both column 1 and 2): Set column temperature to 60°C for 2 minutes, then 10°C/min. to 300°C and hold for 24 min. Set FID Detector to 310°C and injector to 300°C. Conditions may be altered to improve resolution or recovery of petroleum range organics.

7.2.2 Performance Criteria: GC run conditions and columns must be chosen to meet the following criteria:

7.2.2.1 Resolution of C_8 from the solvent front.

7.2.2.2 The column must be capable of separating typical petroleum hydrocarbon components from the surrogates.

7.3 Retention Time Window

7.3.1 Before establishing windows, be certain that the GC system is within optimum operating conditions. Make three injections of the method standard throughout the course of a 72 hour period. Serial injections over less than a 72 hour period result in retention time windows that are too tight.

7.3.2 Calculate the standard deviation of the absolute retention times for the two surrogates, C_8, and C_{40}.

7.3.2.1 The retention time window for individual peaks is defined as a plus or minus three times the standard deviation of the absolute retention time for each component.

7.3.2.2 In those cases where the standard deviation for a particular analyte is zero, the laboratory should use $\pm$0.05 min as a retention time window.

7.3.3 The laboratory must calculate retention time windows for these standards on each GC column and whenever a new GC column is installed. The data are retained by the laboratory.

7.4 PRO Calibration

7.4.1 Initial Calibration – Calibration shall be by external calibration using a minimum of 5 concentration levels for the initial calibration. Quantitation shall be by linear regression.

In all cases, response of the standards must be determined by continuous integration of all responses (excluding surrogates) from a forced baseline beginning at a point prior to the elution of C_8 to a point past C_{40}. All responses must be determined as responses to baseline and not valley to valley. A method is calibrated for all five levels using the area of each of the 17 individual alkanes and the area of the two surrogates and a total area of the Petroleum Hydrocarbon Standard (PRO) (which is the total area of the seventeen alkanes for each level).

7.4.1.1 Linear Regression – The linear regression shall be calculated using the total PRO area versus the PRO concentration. The correlation coefficient shall be equal to or greater than 0.995.

7.4.1.2 The accuracy of the initial calibration shall be verified by injecting a midpoint concentration of a standard mix that has been obtained from a different source. The calculated value shall be within $\pm$ 20% of the expected value.

7.4.2 Continuing Calibration – The calibration curve must be verified on each working day by the injection of a continuing calibration standard (CV) at a midpoint concentration. This standard must be evaluated prior to the analysis of samples.

In addition, a continuing calibration must be run every 10 samples and at the end of the sequence. The concentration of these should vary, with at least one at a level of 1-2 times the calculated PQL as a verification of sensitivity. To accomplish this, continuing calibrations at 50 ug/ml and 20 ug/ml (each component) should be ran.

7.4.2.1 If the concentration of this standard varies from the predicted concentration by more than ± 25%, a new initial calibration curve must be prepared and verified before samples are analyzed.

7.4.2.2 Retention Time Window – Establish daily retention time windows for each analyte of interest using the absolute retention time for each analyte as the midpoint of the window for that day **if** after analyzing the midpoint it is determined that one or more analytes falls outside of the previously established absolute retention time window. The daily retention time window equals the midpoint ± three times the standard deviation determined in Section 7.3.

7.5 Gas Chromatography Analysis

7.5.1 A 1 ul injection volume is analyzed by GC/FID.

7.5.2 If an initial calibration has already been performed, verify the calibration by analysis and evaluation of a mid-point CV on each working day.

In addition, a CV must be run every 10 samples and at the end of the sequence.

7.5.3 Evaluate the CV per 7.4.2.1 and 7.4.2.2. If either performance criteria fails, the instrument must be recalibrated and all samples which were injected after the failed standard must be reanalyzed.

7.5.4 A Methylene chloride blank will be run in every sequence to determine the area generated on normal baseline bleed under the conditions prevailing in the 24 hour period if requested by the client. This area is determined by continuous integration of all responses under the same conditions (i.e. forced baseline and predetermined time interval) as the samples. This blank is calculated as the solvent blank and the value should be less than the PQL.

Methylene chloride blanks should also be run after samples suspected of being highly concentrated to prevent carryover. If the blank analysis shows contamination, additional blanks should be analyzed until the system is shown to be free from contaminants.

7.5.5 If the sample concentration exceeds the linear range of the method in the final extract, the extract must be diluted and reanalyzed.

7.5.6 Baseline correction is allowed to correct for rises due to temperature programming. Range integration is corrected by the automatic subtraction of the baseline established by activation of a programmed run without the injection of any material. Instrument baseline must be established for every batch of samples.

7.6 Calculations

7.6.1 The integrated area for all peaks eluting from n-octane through n-tetracontane shall be determined using a baseline drawn from the baseline point to n-octane to a point past n-tetracontane where the baseline returns to normal. All area including the “hump-a-gram” and surrogate standards shall be included. Do not integrate valley to valley for individual peaks except for the two surrogates. The concentration of the PRO is calculated by using the calibrated curve that is prepared in Target. Target displays a concentration when the file is processed through the appropriate calibrated method.

7.6.2 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

7.6.2.1 Water: Conc (ug/L) = (Amt) (DF) ((Vt/Vo) 1000)

7.6.2.2 Soil/Sediment: Conc (mg/kg) = (Amt) (DF) ((Vt/Vo) (100/(100-M)))

where, Amt = adjusted concentration calculated by Target in ug/ml
Vt = Volume of total extract
Vo = Volume or weight of sample extracted
M = % Moisture
DF = Dilution Factor

7.7 Data Review

7.7.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that

need to be reanalyzed or diluted and reanalyzed. The initial data review is performed in Target Review. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: manual integration.
- Target compound detection: quantitation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next work day. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.8.

7.7.2 Surrogate recovery

The recoveries for o-Terphenyl are compared to the method acceptance limits. The recoveries for n-Triacontane-d_{62} are compared to nominal acceptance limits of 70-130% until laboratory acceptance limits can be established.

The sample is evaluated for recoveries of the surrogate OTP and n-Triacontane-d_{62}. If the recovery is low and there is no apparent matrix effect, the sample should be reanalyzed. If the reanalysis is still low, re-extract. If the recovery is low and there may be a matrix effect, reanalyze to confirm a matrix effect and narrate. If the surrogate is high and the sample results are less than the PQL, or there is likely a matrix effect, narrate.

7.7.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

In Target Review, each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, if the sample contains a concentration of PRO which was integrated “valley to valley”

instead of a "baseline to baseline"), manual integration is performed in Target Review. A "m" qualifier will automatically be printed on the quantitation report summary indicating that a manual integration was performed. For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, Manual Integration, current revision.

7.8 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into KIMS. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the Department Manager for final review. A completed review checklist (Figure 2) is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

See below or refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. It may not be possible to reanalyze samples within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of

the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 For each analytical batch (up to 20 samples), a method blank, laboratory control sample (LCS), matrix spike and matrix spike duplicate or sample duplicate are analyzed. They are carried through all stages of the sample preparation and analysis steps.

8.2 The laboratory shall generate control limits based on +/-3 standard deviations from the average recovery for all spikes and surrogates, and + 3 standard deviations from the average precision value for all duplicates. The limits that area generated must be within the criteria specified in Table 3 below.

8.3 Spike concentrations: The LCS and the MS/MSD are spiked with the seventeen component PRO mix at the same concentration. The spike concentrations are:

	WATER ug/L	SOILS mg/Kg
PRO	850	28.5

The surrogate spike concentrations in the final extract are:

	WATER ug/ml	SOILS mg/kg
o-Terphenyl	50	1.65
n-Triacontane-d_{62}	300	10

8.4 LCS and MS/MSD acceptance criteria and Corrective Action: All QC samples are calculated for percent recovery of the spiked analyte(s). The recoveries are compared to the method acceptance limits. Refer to Table 3 for these limits.
If any spike compound in the laboratory control sample falls outside of the established recovery acceptance limit window, the QC sample is considered to be out of control and any sample that is associated should be reextracted. However, if the recovery is high and the associated samples do not contain the specific compound(s), the data can possibly be accepted with narration.

If a spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable. The cause of the failure is possibly attributable to matrix interference. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.

8.5 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries for o-Terphenyl are compared to the method acceptance limits. The recoveries for n-Triacontane-d_{62} are

compared to nominal acceptance limits of 70-130% until laboratory acceptance limits can be established.

When a sample has a surrogate that falls outside of the method acceptance limit window, the problem should be investigated. If the recovery looks like it is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reextracted

8.6 Non-conformance Report: Whenever data is not acceptable because of a failing LCS or surrogate recovery, a non-conformance report (NCR) must be initiated as soon as possible.

9.0 METHOD PERFORMANCE

9.1 The MDL of this method is estimated to be at least 4 mg/kg for soil and 0.1 mg/L for water. Each laboratory shall establish a laboratory specific MDL for all matrices prior to analyzing any samples.

9.2 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

9.3 Limits of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

9.4 The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the anaysis is not included in the DoD Scope of Accreditation.

9.5 MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

9.6 Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

9.7 Refer to the current revision of the Florida Department of Environmental Protection Method for Determination of Petroleum Range Organics (Method # FL-PRO) for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Florida Department of Environmental Protection, Method for Determination of Petroleum Range Organics, Method # FL-PRO, Revision 1, November, 1995.

ASTM "Standards Methods for Comparison of Waterborne Petroleum Oils by Gas Chromatography," 3328-78.

Wisconsin DNR Modified DRO method, July 1993, Revision 6.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Current version.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-803, Laboratory QA: Self Inspection System, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

LIST OF TABLES AND FIGURES

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Method blank	One per prep batch	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are < PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
LCS	One per prep batch	Method acceptance limits	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are < PQL, narrate. Otherwise, reprep a blank and the remaining samples.
Initial Calibration	Initial cal prior to sample analysis	Correlation coefficient => 0.995	(1) Perform instrument maintenance as needed. (2) Reanalyze and or reprep calibration standards.
CV(At or near the midpoint of the ICAL)	On each working day prior to sample analysis if an ICAL was previously analyzed	± 25 %D	(1) Evaluate the samples: If the %D>+25% and sample results are <PQL, narrate. If %D>±25% and is likely a result of matrix interference, narrate. All samples must be reanalyzed that fall within the standard that exceeded criteria and the last standard that was acceptable.
End of sequence CV	At the end of each 12-hour work shift or after running 10 samples, whichever is sooner	± 25 %D	(1) Evaluate sample data if criteria exceeded due to matrix; narrate, and perform maintenance for new samples. (2) If criteria are exceeded and this is not due to matrix, Reanalyze.
Matrix Spike/Matrix Spike Duplicate	One for every set of 20 samples provided samples aliquots are not depleted	Laboratory established acceptance limits RPD< 20 % for waters and < 25 % for solids	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
Sample Duplicate (If required in lieu of MSD)	One sample duplicate per twenty samples	RPD ≤20 for waters, RPD ≤25 for solids	(1) Evaluate data for matrix interference homogeneity of sample.
Demonstration of analyst proficiency; accuracy and precision	One time per analyst initially and annually thereafter	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

TABLE 1 (cont.)

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Demonstration of analyst proficiency; accuracy and precision	One time per analyst initially and annually thereafter	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file
MDL study/ LOD, LOQ Verifications	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-333-07	FL-PRO, current revision
Apparatus/Materials		
Reagents	5.3 Sodium sulfate purified by vendor 5.5 PRO free sand (Muffled)	7.3 Sodium sulfate purified by heating at 400 deg C for 4 hours or extracting x3 with methylene chloride and drying at 105 deg C. 3.4 Ottawa sand
Sample preservation/ handling		
QC – Method Blank	Table 1 No analyte detected >PQL	10.4 …TRPH value of the blank shall be at or below the established method detection limit.
QC - Surrogates	Use n-Triacontane-$d_{62.}$ Use nominal limits of 70-130 until laboratory limits can be established.	7.4.1 Recommend $C_{39.}$ Use method acceptance limits.
QC - Spikes	8.2 …PRO concentration of 850 ug/L for waters and 28.5 mg/kg for soils.	7.4.4 Total PHS concentration in the spiked sample of 5 mg/L in water or 300 mg/kg in soils. …The concentration of the spike in the sample should be approximately 3-5 times the level expected in the sample…level of the spike should be adjusted…
QC - Accuracy/Precision		
QC – LCS		
QC - MDL		
Procedure	7.4.2.2 Retention Time Window – Establish daily retention time windows for each analyte of interest using the absolute retention time for each analyte as the midpoint of the window for that day **if** after analyzing the midpoint it is determined that one or more analytes falls outside of the previously established absolute retention time window. The daily retention time window equals the midpoint ± three times the standard deviation determined in Section 7.3. 7.5.4 The Methylene chloride blank is analyzed if requested by the client and will be less than the PQL. 7.5.4 Baseline correction is allowed to correct for rises due to temperature programming. Range integration is corrected by the automatic subtraction of the baseline established by activation of a programmed run without the injection of any material. Instrument baseline must be established for every batch of samples. 5.7 The standards are prepared at the following five different concentrations: 200 ug/ml, 100 ug/ml, 50 ug/ml, 20 ug/ml, and 5 ug/ml (per each component).	9.3.2.2 Retention Time Window – The retention time window for the surrogates and C8 and C40 shall be within the established range ... If they are out of acceptance range, a new initial calibration must be prepared and verified before samples are analyzed. 9.5.4 The methylene chloride blank must be analyzed with each sequence and the PRO concentration shall be less than the MDL of the method. 9.5.4 Do not baseline subtract 7.4.3 Suggested calibration levels are 5, 50, 150, 250, 350 and 500 ug/mL of each individual component.

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

TABLE 3

METHOD ACCEPTANCE CRITERIA

	% Recovery			Precision (%RSD)		
	Water	Soil		Water	Soil	
		Soxhlet	Sonication		Soxhlet	Sonication
Matrix Spike Samples	41-101	41-224	62-204	0-20	0-25	0-25
Laboratory Control Spike Samples	55-118	63-135	63-153	0-20	0-25	0-25
Surrogates: OTP	82-142	57-115	62-109			
n-triacontane-D_{62}	70-130	70-130	70-130			

TABLE 4

PQLS

Analyte	Water ug/L	Soil mg/Kg
PRO	500	20

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

FIGURE 1

EXAMPLE OF RUNLOG PAGE

Katahdin Analytical Services, Inc.
Instrument: GC12 (FID)
Amount Injected 1uL

GC Laboratory Instrument Runlog

Method (circle): EPH(MADEP) / FL PRO / TNRCC 1005

Reviewed by/ Date:

DRO/TPH - 8015Mod. / MDEP 4.1.25 / 8100Mod.

Date	Init.	Result File	Sample ID	Y/N	Method	Column	Comments
5-5-11	AC	CEE2012	TB	N	AROB031A	366	
		13					
		14					
		15					
		16	SE2262-1	Y			
		17	TB	N			
		18					
		19					
		20					
		21	SE2316-1	Y			
		22	TB	N			
		23		Y			
		24	ARO50	Y			H2038
			Change Liner / Septa				
5-5-11	AC	25	TB	N	FLPB021A		
		26	TB	Y			
		27	FLP50	Y			H2009
		28	200	Y			H2011
		29	100	Y			H2012
		30	20	Y			H2010
		31	5	Y			H2013
		32	IND	Y			H2014
		33	TB	Y			
		34	WG91154-1	N			
5-6-11		35	-2	N			spike↑
		36	-3	N			↓
		37	AQ LOD	Y			
		38	LOQ	Y			
		39	SE2433-2	N			
		40	-3	N			55↓

GC-007 - Revision 1 - 01/20/2010 QAGC238 0000056

TITLE: **DETERMINATION OF PETROLEUM RANGE ORGANICS (PRO) BY FLORIDA DEPARTMENT OF ENVIRONMENTAL PROTECTION METHOD # FL-PRO**

FIGURE 2

EXAMPLE OF DATA REVIEW CHECKLIST

PRIMARY REVIEW CHECKLIST

Verbal Due Date _____ **(** ☐ **Verbals turned in). Due Date**_____

Client:		Primary	Secondary
Method:		Date:	Date:
SDG No:	Level:	Initials:	Initials:
KAS No:			Approved : ☐ Yes

DODQSM (4.1) ☐ **DOD W/ LAB. LIMITS** ☐ **QUAPP** ☐ **LAB** ☐
(*REPORT* ***ND's to -*** ***POL*** ☐ ***MDL*** ☐ ***LOD*** ☐**)**

List all curves that ***are scanned*** (hard copy not included). _____

_____ _____ _____ _____

Narrate which QC limits were used for (Surr., LCS's MS/MSD's.) ☐
All needed forms are present . ☐
Correct Work Order Number or SDG name (all forms). ☐
Correct project name and spelling (all forms). (Truncated ☐). ☐
Correct file numbers (all forms). ☐
Analysis Date Correct. ☐
Extraction Method & Analysis Method Correct. ☐
Product list compared to ROAs (compounds & PQLs). ☐
Chromatogram reviewed for unlabeled peaks (check product list). ☐
Flagging of all ROAs correct (Florida ☐) (Florida ☐). ☐
All tunes included (level IV) . ☐
All log book pages included (Soil weights,TCLP & SPLP). ☐
Verify DOD QSM criteria and/or Project specific requirements. ☐
Narrate any method deviations. (Blanks, LCS's etc.) ☐
Sign & Date Manual integration **(Narrate as needed).** ☐

Sample I.D's Truncated (**NARRATED**). YES ☐ Please list KAS # below :

_____

QA-044 – Revision 3 – 10/24/2012

TITLE: METHOD FOR DETERMINING VOLATILE PETROLEUM HYDROCARBONS or GASOLINE RANGE ORGANICS (GRO) BY METHOD 8015 (WITH OR WITHOUT MODIFICATIONS)

Prepared By: Peter Lemay Date: 6/97

Approved By:

Group Supervisor: [signature] Date: 5/24/01

Operations Manager: [signature] Date: 5/23/01

QA Officer: Deborah J. Madeau Date: 5.24.01

General Manager: [signature] Date: 9/24/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
02 8015B	Revised sections 7.3.2, 7.4.4, 7.4.1 and added 5.9 to reflect the option of using method 8015 with no modifications - i.e. for South Carolina	DN	5.24.01	5.24.01
03 8015B	Minor editorial changes to sections 7.7.3 and 9.0.	DN	5.22.02	5.22.02
04 8015B	Added definitions and information for new data processing system. Added or changed wording to clarify sections 8+9. Minor changes throughout.	MRC	11.09.04	11.09.04
05 8015B	removed references to TFT added alternate levels for CV's removed Sect. 8.13 (gasoline composite sample) corrected typographical errors and formatting errors	LAD	04/06	04/06
06	Removed all remaining references to TFT and extraction surrogates. Changed wording in Sect. 7.4.2 to reflect current calibration procedure. Added wording to Sect. 7.8.4 clarifying BFB & GRO quantitation. Added wording to Sect. 8.1.2 clarifying Sample Reruns. Added ICV information to Sect. 1, 5, 7 and Table 1	LAD	07/07	07/07

TITLE: METHOD FOR DETERMINING VOLATILE PETROLEUM HYDROCARBONS or GASOLINE RANGE ORGANICS (GRO) BY METHOD 8015 (WITH OR WITHOUT MODIFICATIONS)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
07	Added use of Gasoline Standard for fingerprinting. Minor formatting changes throughout. Edited Section numbers. Clarified Correlation Coefficient Criteria. Added figure for GC Soil prep logbook.	LAD	03/08	03/08
08	Added interference regarding FID. Updated method references. Updated Method Modification Table 2.	LAD	02/09	02/09
09	Update RT window criteria, method blank criteria and "Q" flagging criteria for compliance with DoD QSM version 4.1. Added references.	LAD	08/09	08/09
10	Added Chemstation definition. Changed QC limits to Laboratory statistically derived acceptance limits. Added LOQ, LOD, & MDL information. Updated and added references Updated Figures.	LAD	03/12	03/12

TITLE: METHOD FOR DETERMINING VOLATILE PETROLEUM HYDROCARBONS or GASOLINE RANGE ORGANICS (GRO) BY METHOD 8015 (WITH OR WITHOUT MODIFICATIONS)

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document SOP CA-316-10, titled METHOD FOR DETERMINING VOLATILE PETROLEUM HYDROCARBONS or GASOLINE RANGE ORGANICS (GRO) BY METHOD 8015 (WITH OR WITHOUT MODIFICATIONS).

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document SOP CA-316-10, titled METHOD FOR DETERMINING VOLATILE PETROLEUM HYDROCARBONS or GASOLINE RANGE ORGANICS (GRO) BY METHOD 8015 (WITH OR WITHOUT MODIFICATIONS).

Recipient: _____ Date: _____

TITLE: METHOD FOR DETERMINING VOLATILE PETROLEUM HYDROCARBONS or GASOLINE RANGE ORGANICS (GRO) BY METHOD 8015 (WITH OR WITHOUT MODIFICATIONS)

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the method used by Katahdin Analytical Services technical personnel to measure the concentration of gasoline range organics in water and soil. This procedure measures compounds from MTBE through naphthalene inclusive. This corresponds to a boiling point range between approximately 60°C and 220°C. The analytical procedure is based on a purge-and-trap, Gas Chromatography (GC) procedure.

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (LABORATORY REAGENT BLANK): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix. For soil samples, clean (muffled) sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

GASOLINE RANGE ORGANICS (GRO): All chromatographic peaks eluting from methyl-tert-butylether through naphthalene, inclusive. Quantitation is based on a direct comparison of the area within this range to the total area of the 10 components in the Gasoline Component Standard.

GASOLINE COMPONENT STANDARD: A ten component blend of typical gasoline compounds (Table 4). This standard serves as a quantitation standard and a retention time window for gasoline range organics.

INDEPENDENT CALIBRATION VERIFICATION (ICV): The ICV is obtained from a source external to the laboratory and different from the source of calibration standards. A reagent water blank is spiked with the ICV Standard and analyzed immediately following a calibration.

COMMERCIAL GASOLINE STANDARD: The Commercial Gasoline Standard is analyzed following a calibration and is used for fingerprinting purposes.

LABORATORY CONTROL SAMPLE (GASOLINE COMPONENT SPIKE): A reagent water blank or reagent methanol blank sample spiked with the Gasoline Component Standard and run through the method with samples as a quality control check. The LCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with external prepared test materials.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The MS/MSD is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS/MSD corrected for background concentrations.

METHOD DETECTION LIMIT (MDL): Minimum concentration that an analyte can be measured and reported with a 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. The MDL is determined using EPA Appendix B to Part 136, CFR 40 Ch. 1 (7-1-94) using the Student t Test.

PRACTICAL QUANTITATION LIMIT (PQL): The practical quantitation limit for gasoline is 10 ug/L for water samples and 2.5 mg/Kg for soil samples.

TEMPERATURE BLANK: (If required by project) A vial of water supplied by the laboratory, treated in the same manner as sample vials and carried along with samples, to determine if proper cooling of samples has been achieved (0°C-6°C). A 40 mL or 60 mL vial will be adequate for this purpose.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

PE NELSON TURBOCHROM OR HP CHEMSTATION: data acquisition systems that are used to collect chromatographic data. The systems can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

QUICKFORMS: A laboratory reporting software for Target and Target DB. The QuickForms report module for Target is preconfigured with generalized forms and US EPA CLP report forms and disk deliverables, which can be customized.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in

the analysis of GRO's by Method 8015C (with or without modifications). Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in analysis of GRO's by Method 8015 (with or without modifications) to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in

accordance with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP. Purge vial and methanol waste are disposed of in the "A" waste satellite accumulation area located between the Gasoline Range instruments.

2.0 SUMMARY OF METHOD

The method provides gas chromatographic conditions for the detection of volatile petroleum fractions such as gasoline, Stoddard solvent or mineral spirits. Samples are analyzed utilizing purge-and-trap sample concentration. The gas chromatograph is temperature programmed and utilizes a capillary column (75 meter 0.45mm ID DBVRX or equivalent) to facilitate separation of organic compounds. Detection is achieved by a flame ionization detector (FID) or photoionization detector (PID)/(FID) in series with the non-destructive (PID) first in the series. Quantitation is based on FID detector response to a gasoline component standard utilizing an external standard method.

The method is suitable for the analysis of waters, soils, or wastes. Water samples can be analyzed directly for gasoline range organics by purge-and-trap extraction and gas chromatography. Soil or waste samples are dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanol solution is then analyzed by purge-and-trap GC.

This method is based in part on 1) API's PETROLEUM HYDROCARBON METHODS [Revised August 1993], 2) Wisconsin GRO and DRO methods, 3) SW-846 methods 5030, 8000, and 8015; 4) The Massachusetts TPH methodologies, 5) Maine HETL GRO methods.

3.0 INTERFERENCES

Heavier petroleum products such as diesel fuel may contain some volatile components, producing a response within the retention time range for GRO. Other compounds that respond to the FID such as chlorinated and oxygenated hydrocarbons are detected by this method and will be included in the concentration. If the analyst suspects that compounds are present that are not present in gasoline mixtures, the analyst should suggest additional analysis. Spills of known specific constituents should be analyzed and quantified by a method specific for those constituents.
Samples can become contaminated by diffusion of volatile organics through the sample container septum during shipment and storage or by dissolution of volatiles into the methanol used for preservation. Trip blanks prepared from both reagent water and methanol should be carried through sampling and subsequent storage and handling to serve as a check on such contamination. (Methanol trip blank required only if soil samples

are preserved with methanol in the field.)

Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe and/or purging device must be rinsed between samples with reagent water or solvent. For volatile samples containing high concentrations of water-soluble materials, suspended solids, high boiling compounds or organohalides, it may be necessary to wash the syringe or purging device with a detergent solution, rinse with distilled water, and then dry in a 105°C oven between analyses. The trap and other parts of the system are also subject to contamination, therefore, frequent bake-out and purging of the entire system may be required. A screening step is recommended to protect analytical instrumentation. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of a water blank to check for cross-contamination.

The retention time window definition (methyl-tert-butylether through naphthalene inclusive) introduces a negative bias, however, it improves comparability between laboratory data. Note that gasoline blends often contain 10% ethanol, which could be responsible for a portion of this negative bias.

The flame ionization detector (FID) is a non-selective detector. There is a potential for many non-target compounds present in samples to interfere with this analysis. There is also the potential for analytes to be resolved poorly, especially in samples that contain many analytes. The data user should consider this and may wish to alter the target analyte list accordingly.

4.0 APPARATUS AND MATERIALS

4.1 Gas Chromatograph Analytical system complete with gas chromatograph suitable for purge-and-trap sample introduction and all required accessories, including detectors, column supplies, recorder, gases and syringes. A data system capable of determining peak areas by integrating from baseline to baseline is required.

4.2 Columns (Capillary Columns are Required)

4.2.1 The capillary column must be capable of resolving typical gasoline components. It must be capable of achieving a 60% resolution of all 10 components, with the exception of meta and para-xylene. It must also be capable of separating methyl-tert-butylether from methanol at the concentration resulting from preparation of the standard or in the sample spiked with surrogate. The Resolution is defined as:

$$\frac{\text{(Height of Smaller Peak)(Height of Valley)}}{\text{(Height of Smaller Peak)}} \times 100\ \%$$

4.2.2 Suggested columns: Note either column can be used for soil or water provided the resolution meets the criteria given above.

- DBVRX 75 meter 0.45 mm ID, 2.5 um film thickness (or equivalent).
- RTX 502.2 105 meter 0.53 mm ID, 3.0 um film thickness (or equivalent).
- Any capillary column phase and length and diameter may be used as long as the requirements of resolution of this method are met.

4.3 Detector: Flame ionization (FID), or FID in series with a Photoionization detector (PID).

4.4 Purge-and-trap device: The purge-and-trap device consists of three separate pieces of equipment: the sample purger, the trap, and the desorber. Several complete devices are commercially available.

4.4.1 Commercially available automated sample purge devices may be used, provided that equivalent performance is demonstrated.

4.4.2 The recommended trap is the Supelco H trap which consists of 7.6 cm Carbopack B and 1.3 cm Carbosieve S-III. Refer to the instrument runlog for the trap conditions. The trap length and packing materials may be varied as long as equivalent performance compared to the recommended trap has been verified. Demonstration of equivalency of trap performance must be based on a full range of gasoline components, not just on the 10 components in the standard.

4.4.3 Traps should be conditioned, desorbed, and baked according to the manufacturers' guidelines. The trap may be vented to the analytical column during daily conditioning, however, the column must be run through the temperature program prior to analysis of samples.

4.4.4 The desorber should be capable of rapidly heating the trap to the recommended desorption temperature.

4.5 Analytical balance: A balance capable of accurately weighing 0.0001 g (for preparing standards). A top-loading balance capable of weighing to the nearest 0.01 g (for weighing soil samples).

4.6 Ultrasonic bath.

4.7 VOC Vials: 40 mL VOC vials with Teflon/silicone septa for waters and soils. Soils

not preserved in the field may be collected in wide mouth jars with Teflon lined caps (2 or 4 oz jars recommended).

4.8 Syringes: 5 mL gas-tight syringe (with shutoff valve recommended).

4.9 Syringe valve: Two-way, with luer ends.

4.10 Volumetric flask (class A): 10 mL, 50 mL, 100 mL, 500 mL, and 1,000 mL with a ground-glass or screw-top stopper.

4.11 Microsyringes: 1 uL, 5 uL, 10 uL, 25 uL, 100 uL, 250 uL, 500 uL, and 1,000 uL.

4.12 Disposable pipettes: Pasteur.

4.13 Spatula: Stainless steel.

4.14 40 mL VOA vials with Teflon lined septa or caps for soil extractions performed in the lab.

5.0 REAGENTS

5.1 Reagent Water: Organic free water.

5.2 Methanol: Purge and trap grade or equivalent. Store away from other solvents.

5.3 Acid for preserving water samples: A 1:1 mixture of reagent water and concentrated hydrochloric acid. Use 2 or more drops per 40 mL VOA vial. Acid may be added to the sample at the time of collection or may be added to the vial prior to the collection. Alternatively add 0.1 g of sodium hydrogen sulfate to the empty VOA vial. The final pH of the water should be <2.

5.4 GRO free sand or soil.

5.5 Stock Standards: Purchase individual certified component standards. A concentration of 20 mg/mL is recommended for individual component standards.

5.5.1 Transfer the stock standard solution into a Teflon-sealed screw-cap or crimp cap bottle. Refrigerate, with minimal headspace and protect from light.

5.5.2 Standards must be replaced after 6 months or sooner if comparison with check standards indicates a problem.

5.6 Gasoline Component Standard: Purchase a certified Gasoline Component Standard

from an acceptable supplier like Supelco. These standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation. The component standard must be replaced after 6 months or sooner if comparison with a check standard indicates a problem.

5.7 Calibration Standards: Prepare Calibration standards at a minimum of five concentration levels in reagent water from the Gasoline Component Standard. One of the concentration levels should be at the minimum reporting level. The remaining concentration levels should correspond to the working range of the GC system. Refer to section 7.4.

5.8 Independent Calibration Standard (ICV)/Laboratory Control Spike Standard (LCS) Stock Standards: Purchased form a source external to the laboratory and different from the source of calibration standards. These standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation. The component standard must be replaced after 6 months or sooner if comparison with a check standard indicates a problem.

5.9 Independent Calibration Standard (ICV)/Laboratory Control Spike Standard (LCS) Working Standards: Prepare standards at a concentration level near or at the mid-point of the calibration in methanol from the Independent Calibration Standard (ICV)/Laboratory Control Spike Standard (LCS) Stock Standards.

5.10 Surrogate Control Standard (SCS): The analyst should monitor the performance of the analytical system by spiking each water sample, standard, water blank and diluted soil extract with the surrogate compound Bromofluorobenzene (BFB). All samples are spiked with 2 uL of a 50 ug/mL concentration of BFB to give a final concentration of 20 ug/L.

5.11 Commercial Gasoline: Purchase a certified Commercial Gasoline Fuel Standard from an acceptable supplier like Supelco. This standard should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation. The standard must be replaced after 6 months or sooner if comparison with a check standard indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 Soil core samples may be collected in wide mouth jars when field preservation is not required. Minimum handling is required to reduce VOC loss. Samples that are preserved in the field should be collected in septum vials.

6.1.1 Note on the chain of custody when samples are not preserved in the field. If the sample is not preserved in the field, the container should be filled to

minimize the headspace. If there is a large headspace, this fact should also be noted on the chain of custody.

6.1.2 If samples are not received cold, note this occurrence on the chain of custody.

6.1.3 Refrigerate samples at (4°C ± 2°C) as soon as possible after they are received at the lab.

6.1.4 Analysis must be completed within 14 days of collection.

6.2 Collect water samples in 40 mL septum vials and preserve with acid at time of collection.

6.2.1 Check water samples for air bubble(s) and reject if the bubble(s) are significant.

6.2.2 If samples are not received cold, note this occurrence on the chain of custody.

6.2.3 Place samples in a refrigerator at 4°C ± 2°C as soon as possible after they are received at the lab.

6.2.4 A water trip blank that accompanies all samples and is analyzed with the samples may be required for specific projects.

6.2.5 Water samples must be analyzed within 14 days of collection.

7.0 PROCEDURES

7.1 Volatile compounds are introduced into the gas chromatograph by purge-and-trap. Purge-and-trap may be used directly on groundwater samples. Soils and solids should be analyzed by methanol extraction followed by purge-and-trap. Soil concentrations will be reported on a dry weight basis.

7.2 Gas Chromatography

7.2.1 Conditions for Column 1: Suggested conditions for a 75 m x 0.45 mm I.D. DBVRX, or equivalent: Set helium column pressure as recommended by the manufacturer. Set column temperature to 35°C for 7 min, then 4°C/min to 130°C, then 8°C/min to 160°C, 20°C/min to 230°C (hold for 4 min). Conditions may be altered to improve resolution of gasoline range organics.

7.2.2 Other columns: Set GC conditions to meet the criteria in Section 4.2.1.

7.3 Retention Time Window and Quantitation

7.3.1 Gasoline Range Organics (GRO): All chromatographic peaks eluting from methyl-tert-butylether through naphthalene, inclusive. Quantitation is based on a direct comparison of the area within this range to the total area of the 10 components in the Gasoline Component Standard. (Using a "baseline to baseline" integration as opposed to a "valley to valley" integration.)

Note: The area of the MTBE peak may be determined by tangent skimming when necessary.

7.3.2 The retention time window is defined as beginning approximately 0.1 minutes before the retention time of methyl-tert-butylether and ending 0.1 minutes after the retention time of naphthalene in the calibration run.

Please note that using the nominal window of 0.1 minutes and the use of the retention time markers methyl-tert-butylether and naphthalene may not be allowable for certain states, federal programs, or clients. For projects requiring DoD QSM, current version,, the retention time width is plus or minus three times the standard deviation for each analyte retention time from a 72-hour study. South Carolina only allows the use of established retention time windows and the retention time markers 2-Methylpentane and 1,2,4-Trimethylbenzene. The retention time windows must be established as described below.

7.3.2.1 Three injections of the GRO component standard mix are made throughout the course of a 72 hour period.

7.3.2.2 The standard deviation of the three retention times for 2-Methylpentane and 1,2,4-Trimethylbenzene are calculated.

7.3.2.3 Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window.

7.3.2.4 Retention time windows are calculated for each standard on each GC column and whenever a new GC column is installed. The data is kept on file in the laboratory.

7.3.2.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply a nominal window of 0.03 minutes.

7.3.3 The laboratory must determine retention time windows for the first and last

standard on each GC column and whenever a new GC column is installed. This data must be retained by the laboratory.

7.3.4 Quantify by summing all peak areas eluting from methyl-tert-butylether through naphthalene, inclusive.

7.4 GRO Calibration

7.4.1 Run the Gasoline Component Standard at a minimum of five concentration levels at the minimum reporting level and covering the working range of the instrument. When the calibration curve is run an independent check standard should also be run to validate the curve. A gasoline standard is also run with the calibration curve for fingerprinting purposes.

Please note that using the Gasoline component calibration standard may not be allowable for certain states, federal programs, or clients. South Carolina requires the use of a fuel oil as the calibration standard. If the specific fuel type is known, the laboratory should obtain a pure sample of material from the tank that is leaking. For unknown samples, the instrument is calibrated using Gasoline fuel. The calibration levels for Gasoline are prepared at the same concentrations as that of the GRO calibration. The 100 ug/L standard is used as the mid-point or calibration verification standard (CV).

7.4.2 The instrument is calibrated by injecting a specific amount of one of the three mixes as indicated in the table below. Mix A contains the GRO component mix at 25 ug/mL and the surrogate BFB at 5 ug/mL. Mix B contains the GRO component mix at 250 ug/mL and the surrogate BFB at 50 ug/mL. Mix C contains the GRO component mix at 2500 ug/mL and the surrogate BFB at 500 ug/mL.

Amount injected	Concentration of GRO ug/L	Concentration of BFB ug/L
2 uL of Mix A	10	2
2 uL of Mix B	100	20
5 uL of Mix B	250	50
10 uL of Mix B	500	100
2 uL of Mix C	1000	200
4 uL of Mix C	2000	400

The solutions are added directly in the 5 mL glass syringe as follows:

- Add the aliquot of calibration solution directly to the reagent water in the glass syringe by inserting the needle through the syringe end.

- When discharging the contents of the microsyringe, be sure that the end of the syringe needle is well beneath the surface of the reagent water.

- Inject the standard into the purge vessel through the two-way valve.

7.4.3 Inject 5 mLs of each calibration standard utilizing the purge-and-trap analysis. Retention time window position shall be set using the midpoint standard from the curve. Tabulate the entire peak area (baseline to baseline) for the ten components against the mass injected. The results are used to prepare a calibration curve by linear regression.

7.4.4 An Independent Calibration Verification Standard is analyzed immediately after calibration, before any samples are analyzed. For projects requiring DoD QSM, current version,, all project analytes must fall within the established retention time windows.

7.4.5 A gasoline standard is analyzed following the calibration curve to be used for fingerprinting purposes.

7.4.6 The working calibration curve must be verified at the beginning and end of each analytical sequence by the injection of a mid-point Gasoline Component Standard or calibration verification standard (CV). A CV must also be analyzed every 10 samples or after a 12 hour shift, whichever is sooner. If the response for the CV varies from the predicted response by more than 20%, the analytical system should be examined to determine the cause and corrective action should be performed and/or a new CV should be prepared and analyzed. If the 20% criteria is still not met, the system must be recalibrated. All samples must be reanalyzed that fall within the standard that exceeded criteria and the last standard that was acceptable.
The closing CV should be at a level different than the opening CV.

Please note that using the Gasoline component calibration verification standard (CV) as well as an acceptance criteria of 20% may not be allowable for certain states, federal programs, or clients. For projects requiring DoD QSM, current version,, all project analytes must fall within the established retention time windows. South Carolina requires the use of a fuel oil as the CV. Also, South Carolina requires the analysis of a standard that contains 2-Methylpentane and 1,2,4-Trimethylbenzene at the beginning and end of each 12 hour work shift. The acceptance criteria for the fuel oil CV is 15% and 2-Methylpentane and 1,2,4-Trimethylbenzene must fall within the established retention time window.

7.5 Gas Chromatographic Analysis of Water Samples

Please refer to Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique”, current revision, for more information on subsampling.

7.5.1 Introduce volatile compounds into the gas chromatograph using the purge-and-trap method. For automated systems follow the manufacturers’ recommended procedure.

7.5.2 Adjust the purge gas flow rate (nitrogen or helium) to 25-40 mL/min on the purge-and-trap device.

7.5.3 Remove the plunger from a 5 mL gas-tight syringe that has been rinsed three times with reagent water. Open the sample or standard bottle and carefully pour the sample into the syringe. Replace the plunger and vent any residual air while adjusting the sample volume to 5 mL. Care must be taken to prevent air from leaking into the syringe.

7.5.4 For samples requiring dilutions, the sample may be diluted directly in the 5 mL gas-tight syringe. Alternatively, the following procedure is appropriate for diluting purgeable samples. All steps must be performed without delays until the diluted sample is in a gas-tight syringe.

7.5.5 Dilutions may be made in volumetric flasks (10 mL to 100 mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for highly concentrated samples.

7.5.6 Calculate the approximate volume of reagent water to be added to the volumetric flask selected and add slightly less than this volume of reagent water to the flask.

7.5.7 Inject the proper aliquot of samples (taken from a filled VOA vial or from a storage syringe prepared as in Section 7.5.3) into the flask. If aliquots of less than 1 mL are required, use microliter syringes and deep injections to transfer the sample aliquot to the flask. Dilute the sample to the mark with reagent water. Cap the flask and invert three times. Repeat the above procedure for additional dilutions. Alternatively the dilutions can be made directly in a gas tight syringe to avoid further loss of volatiles.

7.5.8 Fill a 5 mL syringe with diluted sample as in Section 7.5.3.

7.5.9 For aqueous LCSs, use a 5 mL syringe rinsed three times with reagent water. The volume is then brought to 5 mL and 2 uL of a 50 ug/mL concentration of BFB and 2 uL of a 250 ug/mL concentration of the GRO

LCS Mix are added to the 5 mL syringe. For the MS and/or MSD, after rinsing with water, the syringe is rinsed with sample and then is brought to 5 mL with sample. The same amount of the same standards used for the LCS are added to the 5 mL syringe of sample.

7.5.10 Add surrogate to sample or diluted sample by spiking 2 uL of a 50 ug/mL solution of BFB directly into the syringe. Inject sample into the purging chamber.

7.5.11 Close the valve and purge the sample for 11 minutes at ambient temperature.

7.5.12 At the conclusion of the purge time, the purge and trap concentrator should wait in desorb ready mode until the GC temperature stabilizes at 35°C. The purge-and-trap will then be triggered by the GC as it initiates its run and the concentrator will move automatically into the desorb mode. As desorption is initiated the trapped materials are introduced to the gas chromatographic column by rapidly heating the trap to the manufacturer's recommended desorption temperature and backflushing the trap for the recommended desorption time.

7.5.13 Wash the ALS position with a minimum of one 5 mL flush of reagent water (or methanol followed by reagent water) to avoid carryover of pollutant compounds into subsequent analyses.

7.5.14 After desorbing the sample, recondition the trap by baking the purge-and-trap device at the recommended bake temperature. After approximately 7-35 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

7.5.15 If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. When a sample is analyzed that has a saturated response from a compound, this analysis must be followed by a blank reagent water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences.

7.5.16 All dilutions should keep the response of the major constituents (previously saturated peaks) in the linear range of the curve.

7.5.17 All water samples should be checked to make sure that they were preserved. After sample analysis (or after removing an aliquot for analysis), check the pH of the water using pH paper. If the pH is not <2, this fact

should be noted in the instrument run-log, and noted in the sample narrative.

7.6 Methanol Extraction for Soil/Sediment

7.6.1 This method is based on extracting the sediment/soil with methanol. An aliquot of the extract is added to reagent water and purged at the conditions indicated in Table 3.

7.6.1.1 Add approximately 10 g of sample into a tared sample vial and weigh to the nearest 0.01 g. Immediately add 10 mLs of methanol to the vial.

7.6.1.2 If the soil sample has been methanol preserved in the field, both the weight of the soil and the volume of the methanol must be recorded. The ratio of the soil to methanol should be approximately 1 gram soil: 1 milliliter of methanol with a minimum soil weight of 10 grams.

7.6.1.3 For the LCS/LCSD, 10 g of sand is added to a 40 mL VOA vial followed by the addition of 10 mL of methanol. Then 12.5 uL of a 2000 ug/mL concentration of Gasoline Component Spike solution is spiked into the methanol.

7.6.1.4 The MS/MSD is prepared by adding 10 g of sample into a 40 mL VOA vial followed by 10 mL of methanol. Then 12.5 uL of a 2000 ug/mL concentration of Gasoline Component Spike solution is spiked into the methanol.

7.6.1.5 Record all weights and volumes in the GC Soil Prep Logbook (Figure 1).

7.6.2 Shake the sample(s) and QC for 2 minutes and sonicate for 20 minutes.

7.6.3 Allow sediment to settle until a layer of methanol is apparent. Centrifuge if necessary. If not analyzed immediately store at 6°C or less.

7.6.4 Using a microliter syringe, withdraw an appropriate volume.

7.6.5 Remove the plunger from a 5 mL gas tight syringe equipped with a syringe valve that has been rinsed three times with reagent water and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Pull the plunger to 5 mLs and add 20 uL of the sample extract.

7.6.6 Add surrogate to sample or diluted sample by spiking 2 uL of a 50 ug/mL solution of BFB directly into the syringe. Inject sample into the purging chamber.

7.6.7 Proceed with the analysis as in 7.5.11-7.5.16. Analyze all reagent blanks and QC samples on the same instrument as that used for the samples. The reagent blank should contain 20 uL of the methanol used to extract the samples (or a volume equal to the largest amount of extract purged).

7.6.8 If the responses exceed the calibration or linear range of the systems, repeat the analysis using a smaller aliquot of methanol extract.

7.7 Calculations

7.7.1 GRO Calibration: The concentration of Gasoline Range Organics in the sample is determined from a summation of the total peak area for all chromatographic peaks eluting from methyl-tert-butylether through naphthalene, inclusive, using the calibration curve. (Using a "baseline to baseline" integration as opposed to a "valley to valley" integration.) Refer to Section 7.3 (Retention Time Windows and Quantitation).

Quantitation may be based on a linear regression equation derived from the calibration curve.

Linear regression: From the calibration standards GC responses, R, and their known concentrations, C in ug/L, the following linear equation may be derived [R is plotted on the y axis; C is plotted on the x axis]:

$$R = mC + b; \text{ which can be rearranged to } C = (R - b)/(m).$$

Using the slope (m) and the intercept (b) from this equation the concentration of the sample can be calculated.

7.7.2 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration when the file is processed through the appropriate calibrated method.

7.7.3 The concentrations from the reports are then incorporated to arrive at a final sample concentration.

Water: Concentration (ug/L) = (C) (5mL)/(Vs)

Soil: Concentration (mg/Kgdrywt) = (C) (V_m)(0.005L)/(Ws)(V_E)(D)

Where: C = Concentration calculated by Turbochrom in ug/L
Vs = Volume of sample purged in mL
Ws = Weight of sample extracted in grams
V_M = Volume of methanol in mL
V_E = Volume of extract purged in mL
D = Decimal total solids

7.7.4 Blank areas may not be subtracted from sample areas. Chromatographic baseline rises due to temperature programming may be corrected for by using baseline correction. The baseline correction may be performed by the most convenient method that the data handling system allows.

7.7.5 If an aqueous method blank exceeds the PQL of 10 ug/L, all water samples associated with this blank must be rerun. However, if the sample concentration exceeds the blank contamination by 10 times, the contribution from the blank is negligible, and the data may be reported with narration. For projects requiring DoD QSM, current version,, the method blank must not detect any analytes >1/2 the PQL and >1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).

7.7.6 If a methanol blank exceeds the PQL of 2.5 mg/Kg, all soil samples associated with this blank must be rerun. However, if the sample concentration exceeds the blank contamination by 10 times, the contribution from the blank is negligible, and the data may be reported with narration. For projects requiring DoD QSM, current version,, the method blank must not detect any analytes >1/2 the PQL and >1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).

7.7.7 To ensure that peaks outside the GRO window are not missed, run the chromatogram out 5 minutes past the last component in the GRO component standard. All significant peaks (and baseline rises) outside the window should be qualitatively assessed.

7.8 Data Review

7.8.1 Initial Data Review

7.8.1.1 The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed.

The initial data review is performed on the detailed quantitation reports of the analyzed samples. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or reextracted. These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery.
- Chromatography: manual integration.
- Target compound detection: quantitation, false positives.

7.8.1.2 The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next work day. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.9.

7.8.2 Surrogate recovery

7.8.2.1 All recoveries must meet the most recent laboratory established acceptance limits, which are listed on the GC Laboratory Surrogate Acceptance Limit sheet.

7.8.2.2 The sample is evaluated for recovery of the surrogate BFB for water samples and soil samples. For both water and soil samples, if the recovery of BFB is low and there is no apparent matrix effect, the sample should be reanalyzed. If the BFB is low and there may be a matrix effect, reanalyze to confirm a matrix effect. If the surrogate is high and the sample results are less than the PQL, or there is likely a matrix effect, narrate.

7.8.2.3 For projects requiring DoD QSM, current version,, Q-flag all detected analytes in the sample if the surrogates fail the acceptance criteria.

7.8.3 Chromatography

7.8.3.1 The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries.

7.8.3.2 Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating

the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

7.8.3.3 Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Any manual integrations that are necessary (for instance, if the sample contains a concentration of GRO which was integrated "valley to valley" instead of a "baseline to baseline"), are performed in Target Review. An "M" qualifier will automatically be printed on the quantitation report summary. For specific procedures on how to manually integrate, refer to Katahdin SOP QA-812, Manual Integration, current revision.

7.8.4 Target Compound Detection

To correctly quantitate the GRO concentration, the area of the surrogate BFB is not included in the GRO timed range area. In some instances, BFB may need to be manually integrated in Target Review to correctly quantitate the BFB and GRO concentrations.

7.9 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 for a summary of QC requirements, acceptance criteria, and corrective actions. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are

based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Due to time constraints, samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 The analyst must make an initial demonstration of the ability to generate acceptable accuracy and precision with this method by successful analysis of the following:

8.1.1 Replicate Commercial Gasoline Spikes in Water: Analysis of 4 replicates at a concentration of 100 ug/L (in water) with an accuracy falling between 60% and 140% of the known concentration with a precision of 20% or less.

8.1.2 Replicate Commercial Gasoline Spikes in Soil: Analysis of 4 replicates at a concentration of 25 mg/Kg with an accuracy between 60% and 140% of the known concentration and the precision should be within 20%. Soil spikes should be prepared and analyzed as described in Section 7.6.1.3.

8.2 For every 20 samples analyzed the lab must analyze a set of duplicate Gasoline component Spikes in water. The duplicate spikes must be run through the method in the same manner as samples. The accuracy of the two water spikes must be within the laboratory's statistically derived acceptance limits. and the percent relative difference between the two values must be 20% or less.

Projects requesting DoD QSM, Current Version, require Q-flagging the specific LCS analytes that fail and are detected in the associated samples. MS/MSD failures require a J-flag in the parent sample for the analytes that fail the acceptance criteria.

8.3 With every analytical sequence containing soil samples, the lab must analyze one Gasoline Component Spike in clean sand or soil. The spike amount should fall in the linear range of the detector. The spike recovery must be within the laboratory's statistically derived acceptance limits. If soils are extracted in the lab, the soil spike should be prepared at the time of the extraction and analyzed along with the samples.

Projects requesting DoD QSM, Current Version, require Q-flagging the specific LCS analytes that fail and are detected in the associated samples. MS/MSD failures require a J-flag in the parent sample for the analytes that fail the acceptance criteria.

8.4 A water blank (containing purge-and-trap surrogate) must be run with every analytical sequence containing water samples, utilizing the same water used to prepare standards and make dilutions. The amount of material in the blank should not be at or above the PQL. For projects requiring DoD QSM, Current Version,, the method blank must not detect any analytes >1/2 the PQL and >1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).

8.5 A reagent methanol blank (containing purge-and-trap surrogate) prepared with clean (muffled) sand must be run with each analytical sequence containing soil samples, utilizing the same methanol used to prepare standards and dilute extracts and a methanol spike of 20 uL (or largest amount of extract used). The amount of material in the blank should not be at or above the PQL. For projects requiring DoD QSM , currnt version,, the method blank must not detect any analytes >1/2 the PQL and >1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).

8.6 Calibration requirements

8.6.1 When linear regression analysis is used for calculations the correlation coefficient (r) must be at least 0.995. For linear models, Target reports r^2. This is calculated by either calculating r or squaring the result or by calculating the coefficient of determination. For a linear calibration, the equation for either is the same. The value for r^2 must greater than or equal to 0.990.

8.6.2 When average RF is used for calculations, the relative standard deviation of the RF's must not exceed 20%.

8.6.3 An Independent Calibration Verification Standard (ICV) (obtained from a source independent of the calibration standards) should be analyzed concurrent with the calibration standards in order to confirm the validity of the calibration curve. The ICV should fall within 20% of the expected value using the calibration data.

8.6.4 The calibration curve must be verified with each analytical sequence by running an opening and closing Calibration Verification Standard (CV). The closing CV is at 250 ppb. A CV must also be analyzed every 10 samples or after a 12 hour shift, whichever is sooner. The response must fall within 20% of the expected response.

8.7 If any of the criteria above are not met, the problem must be corrected before further

samples are analyzed. Any samples that were analyzed following standards that did not meet calibration criteria must be reanalyzed (if reanalysis is not possible, the data must be flagged.)

8.8 Laboratory Spiked Duplicates are to be run at a frequency of 10%.

Alternatively duplicate samples and spiked samples can be substituted for the laboratory spiked duplicates at a frequency of 10%. Care must be taken to ensure that the samples are homogeneous before analyzing duplicates and spikes.

8.9 One methanol field blank must accompany each sampling event (for each site and each day that samples are collected and preserved in the field).

8.10 Trip blanks, field blanks, field duplicates and/or matrix spikes may be required for specific sampling programs.

8.11 Water blanks should be run after samples suspected of being highly concentrated to prevent carryover.

8.12 It is recommended that an acceptance criteria be established for recoveries of surrogates. Collect recoveries from 30 samples where no interference is suspected and calculate the mean recovery (X) and standard deviation (S). The acceptance limits for samples not exhibiting matrix interference will be X-3S to X+3S. The warning limits will be X-2S to X+2S. Plotting the surrogate recoveries on a control chart will make checking recoveries easier and is highly recommended. Refer to the GC Laboratory Surrogate Acceptance Limits sheet for the current limits used.

If surrogate recovery is outside of the established limits, verify calculations, dilutions, and standard solutions. Verify instrument performance, including checking for leaks and purge problems if the recovery is low. Low recovery may be due to the sample matrix. The analysis should be repeated, if the recovery is less than 50% or, if the analyses cannot be repeated, the data should be flagged.

High recoveries may be due to coeluting matrix interference. Surrogate recoveries may be reported as "masked" in high level samples exhibiting matrix interference. These samples do not need to be rerun solely to try to bring surrogate recovery into acceptance limits.

For projects requiring DoD QSM, current version,, Q-flag all detected analytes in the sample if the surrogates fail the acceptance criteria.

8.13 Non-conformance Report (NCR): Whenever data is not acceptable because of a failing LCS or surrogate recovery, a non-conformance report must be initiated as soon as possible.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Detection (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 8015 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods", SW-846, third Edition, Final Update IV, February 2007, Method 8000B, 8015C, and 5030B.

Wisconsin DNR Modified GRO, July 1993

American Petroleum Institute, "Method for Determination of Gasoline Range Organics", 9/93.

Maine HETL, Method 4.2.17, Modified Method for the Determination of GROs, 9/95.

TITLE: METHOD FOR DETERMINING VOLATILE PETROLEUM HYDROCARBONS or GASOLINE RANGE ORGANICS (GRO) BY METHOD 8015 (WITH OR WITHOUT MODIFICATIONS)

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), current version.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

LIST OF TABLES AND FIGURES

TITLE: METHOD FOR DETERMINING VOLATILE PETROLEUM HYDROCARBONS or GASOLINE RANGE ORGANICS (GRO) BY METHOD 8015 (WITH OR WITHOUT MODIFICATIONS)

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
GRO by Method 8015	Method blank	One per twenty samples or every day whichever is more frequent.	GRO not detected ≥PQL For projects requiring DoD QSM, current version,, the method blank must not detect any analytes >1/2 the PQL and >1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater).	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are < PQL or > 10X the blank concentration. Otherwise, reprep a blank and the remaining samples.
	LCS/LCSD	One set per twenty samples or every day whichever is more frequent.	Laboratory established acceptance limits	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was analyzed and acceptable, narrate. If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS recovery is high but the sample results are < PQL, narrate. Otherwise, reprep a blank and the remaining samples.
	ICAL consists of a minimum of 5 calibration standards. The low standard is at or near the PQL.	Once prior to initiating method then as needed (when CV fails).	Correlation coefficient (r) ≥ 0.995, (r^2) ≥ 0.990. See sect. 8.6.1 for further information.	(1) Perform instrument maintenance as needed and reprep and/or reanalyze the 5 calibration standards.
	CV at or near the mid-point of the calibration curve.	Beginning of each analytical sequence if ICAL previously run; end of analytical sequence; every 10 samples or after 12 hour shift, whichever is sooner	+/- 20% D	(1) Evaluate the samples: If the %D>±20% and sample results are <PQL, narrate. (2) If %D>±20% at the end of a sequence and is likely a result of matrix interference (i.e. samples analyzed before it had bad matrices), evaluate the samples between the opening and closing CV and narrate as needed. All samples must be reanalyzed that fall within the standard that exceeded criteria and the last standard that was acceptable.

TITLE: METHOD FOR DETERMINING VOLATILE PETROLEUM HYDROCARBONS or GASOLINE RANGE ORGANICS (GRO) BY METHOD 8015 (WITH OR WITHOUT MODIFICATIONS)

TABLE 1, cont'd

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
GRO by Method 8015	Matrix Spike/Matrix Spike Duplicate	One set for every twenty samples For projects requiring DoD QSM, current version,, one set per preparatory batch, per matrix	Laboratory established acceptance limits RPD $\leq$30	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable, reprep the samples and QC.
	Sample Duplicate (If required in lieu of MSD)	One sample duplicate per twenty samples	RPD $\leq$20	(1) If lab QC in criteria and matrix interference suspected, flag data (2) Else, reanalyze
	Demonstration of analyst proficiency; accuracy and precision	One time per analyst initially and annually thereafter	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file
	MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: METHOD FOR DETERMINING VOLATILE PETROLEUM HYDROCARBONS or GASOLINE RANGE ORGANICS (GRO) BY METHOD 8015 (WITH OR WITHOUT MODIFICATIONS)

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-316-10	Method 8015, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures	7.3.1 Gasoline Range Organics (GRO): All chromatographic peaks eluting from methyl-tert-butylether through naphthalene, inclusive. **(modify for non-South Carolina clients only)**	7.4.2 Two specific gasoline components are used to establish the range, 2-methylpentane and 1,2,4-trimethylbenzene.
Procedures	7.4.1 A gasoline component standard is analyzed instead of a commercial fuel standard. **(modify for non-South Carolina clients only).**	7.3.3 …use recently purchased commercially available fuel
QC Method Blank		
QC Accuracy/Precision		

TITLE: METHOD FOR DETERMINING VOLATILE PETROLEUM HYDROCARBONS or GASOLINE RANGE ORGANICS (GRO) BY METHOD 8015 (WITH OR WITHOUT MODIFICATIONS)

TABLE 3

PURGE-AND-TRAP OPERATING PARAMETERS

RECOMMENDED CONDITIONS FOR SUGGESTED TRAP	
Purge gas	Nitrogen or Helium
Purge gas flow rate (mL/min)	40
Purge time (min)	11.0 $\pm$ 0.1
Purge temperature (°C)	40°C or less
Desorb temperature (°C)	250°C

TITLE: METHOD FOR DETERMINING VOLATILE PETROLEUM HYDROCARBONS or GASOLINE RANGE ORGANICS (GRO) BY METHOD 8015 (WITH OR WITHOUT MODIFICATIONS)

TABLE 4

GASOLINE STANDARD COMPONENTS AND CONCENTRATIONS

COMPONENT	**CONCENTRATION, ug/mL**
Methyl-tert-butylether (MTBE)	1000
Benzene	1000
Toluene	1000
Ethylbenzene	1000
m-Xylene	1000
p-Xylene	1000
o-Xylene	1000
1,2,4-Trimethylbenzene	1000
1,3,5-Trimethylbenzene	1000
Naphthalene	1000
TOTAL	10,000

TABLE 5

PQLs

Analyte	**Water ug/L**	**Soil mg/Kg**
GRO	10	2.5

TITLE: METHOD FOR DETERMINING VOLATILE PETROLEUM HYDROCARBONS or GASOLINE RANGE ORGANICS (GRO) BY METHOD 8015 (WITH OR WITHOUT MODIFICATIONS)

FIGURE 1

GC SOIL PREP LOGBOOK

KATAHDIN ANALYTICAL SERVICES
GC SOIL PREP LOG

Date of Sample Preparation	Analyst Initials	Sample #	Sample Weight (g)	Volume (mL) Methanol ☑ or DI Water ☑	Spike ID and Volume (µL)	Surrogate ID and Volume (µL)	Method	Comments
2-17-12	EKL	WG104863-1	10.00	10 mL MeOH	—	—	8015M-GRO 4.2.17	
↓	↓	-2	9.99	↓	12.5 uL AMP3120 @ 2000 ug/mL	—	↓	
↓	↓	-3	10.02	↓	↓	—	↓	
↓	↓	SF0324-15	5.00	5 mL*	—	—	↓	NaSC PE 1 mL wt 5055→10mL DI
↓	↓	SF0437-3	5.00	5 mL MeOH	50 uL UST6222m SL lot #6047-08	—	↓	
2-22-12	EKL	WG104971-1	15.00	15 mL MeOH	NA	70 uL AMP3245 @ 5000 ug/mL	MA-VPH	R187899
↓	↓	-2	15.02	↓	35 uL AMP3099 @ 10,000 ug/mL	NA	↓	
↓	↓	-3	15.01	↓	↓	↓	↓	
↓	↓	SF0785-1	18.48	↓	NA	70 uL AMP3245 @ 5000 ug/mL	↓	
↓	↓	-2	19.49	↓	↓	↓	↓	
↓	↓	-3	16.45	↓	↓	↓	↓	
↓	↓	SF0804-1	17.52	↓	↓	↓	↓	
↓	↓	SF0843-1	15.16	↓	↓	70 uL AMP3246 @ 5000 ug/mL	↓	
↓	↓	SF0847-3	8.54	↓	↓	↓	↓	
↓	↓	↓ -4A	8.24	↓	↓	↓	↓	green
↓	↓	↓ -4B	6.48	↓	↓	↓	↓	pink

GC-012 – Revision 1 – 08/18/10

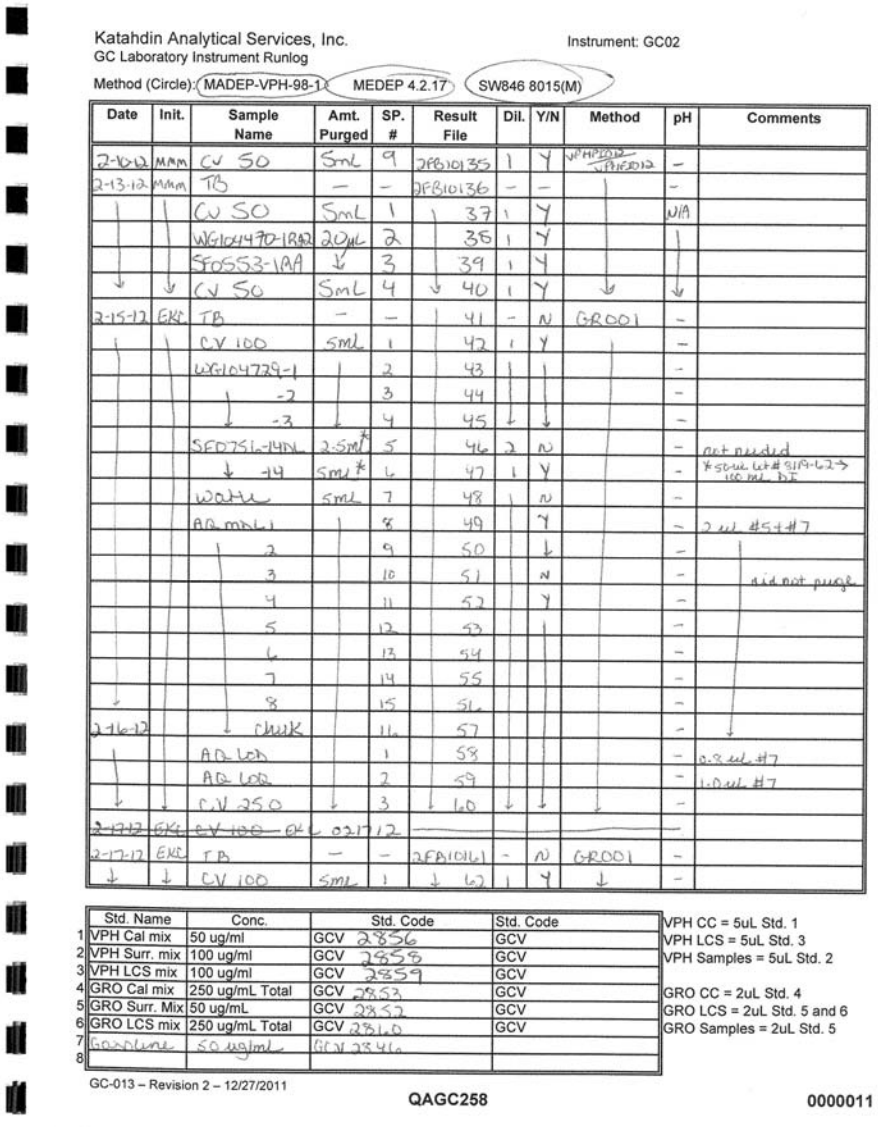
QAGC256

0000005

TITLE: METHOD FOR DETERMINING VOLATILE PETROLEUM HYDROCARBONS or GASOLINE RANGE ORGANICS (GRO) BY METHOD 8015 (WITH OR WITHOUT MODIFICATIONS)

FIGURE 2

INSTRUMENT RUNLOG PAGE



Katahdin Analytical Services, Inc.
GC Laboratory Instrument Runlog

Instrument: GC02

Method (Circle): MADEP-VPH-98-1 | MEDEP 4.2.17 | SW846 8015(M)

Date	Init.	Sample Name	Amt. Purged	SP. #	Result File	Dil.	Y/N	Method	pH	Comments
2-16-12	MMM	CV 50	5mL	9	2FB10135	1	Y	VPHPIONA-JPHEDI2	–	
2-13-12	MMM	TB	–	–	2FB10136	–	–		–	
		CV 50	5mL	1	37	1	Y		N/A	
		WG104970-1RE2	20µL	2	38	1	Y			
		SF0553-1AA		3	39	1	Y			
		CV 50	5mL	4	40	1	Y			
2-15-12	EKE	TB	–	–	41	–	N	GRO01	–	
		CV 100	5mL	1	42	1	Y		–	
		WG104729-1		2	43				–	
		-2		3	44				–	
		-3		4	45				–	
		SF0756-14NL	2.5mL*	5	46	2	N		–	not needed *stock wt#31A-623 100 mL DF
		-14	5mL*	6	47	1	Y		–	
		water	5mL	7	48	1	N		–	
		AB mix 1		8	49		Y		–	2 uL #5+#7
		2		9	50				–	
		3		10	51		N		–	did not purge
		4		11	52		Y		–	
		5		12	53				–	
		6		13	54				–	
		7		14	55				–	
		8		15	56				–	
2-16-12		CHUK		16	57				–	
		AB LOA		1	58				–	0.5 uL #7
		AB LOB		2	59				–	1.0 uL #7
		CV 250		3	60				–	
~~2-17-12~~	~~EKE~~	~~CV 100~~	~~OK~~	02/17/12					–	
2-17-12	EKE	TB	–	–	2FB10161	–	N	GRO01	–	
		CV 100	5mL	1	62	1	Y		–	

	Std. Name	Conc.	Std. Code	Std. Code
1	VPH Cal mix	50 ug/ml	GCV 2856	GCV
2	VPH Surr. mix	100 ug/ml	GCV 2858	GCV
3	VPH LCS mix	100 ug/ml	GCV 2859	GCV
4	GRO Cal mix	250 ug/mL Total	GCV 2853	GCV
5	GRO Surr. Mix	50 ug/mL	GCV 2852	GCV
6	GRO LCS mix	250 ug/mL Total	GCV 2860	GCV
7	Gasoline	50 ug/mL	GCV 2846	
8				

VPH CC = 5uL Std. 1
VPH LCS = 5uL Std. 3
VPH Samples = 5uL Std. 2

GRO CC = 2uL Std. 4
GRO LCS = 2uL Std. 5 and 6
GRO Samples = 2uL Std. 5

GC-013 – Revision 2 – 12/27/2011

QAGC258

0000011

TITLE: METHOD FOR DETERMINING VOLATILE PETROLEUM HYDROCARBONS or GASOLINE RANGE ORGANICS (GRO) BY METHOD 8015 (WITH OR WITHOUT MODIFICATIONS)

FIGURE 3

PRIMARY REVIEW CHECKLIST

PRIMARY REVIEW CHECKLIST

Verbal Due Date _____ **(** ☐ **Verbals turned in). Due Date**_____

Client:		Primary	Secondary
Method:		Date:	Date:
SDG No:	Level:	Initials:	Initials:
KAS No:			Approved : ☐ Yes

DODQSM (4.1) ☐ **DOD W/ LAB. LIMITS** ☐ **QUAPP** ☐ **LAB** ☐
(*REPORT* ***ND's*** *to -* ***POL*** ☐ ***MDL*** ☐ ***LOD*** ☐**)**

List all curves that ***are scanned*** (hard copy not included). _____

_____ _____ _____ _____

Narrate which QC limits were used for (Surr., LCS's MS/MSD's.) _____

All needed forms are present . _____

Correct Work Order Number or SDG name (all forms). _____

Correct project name and spelling (all forms). (Truncated ☐). _____

Correct file numbers (all forms). _____

Analysis Date Correct. _____

Extraction Method & Analysis Method Correct. _____

Product list compared to ROAs (compounds & PQLs). _____

Chromatogram reviewed for unlabeled peaks (check product list). _____

Flagging of all ROAs correct (Florida Flagging ☐). _____

All tunes included (level IV) . _____

All log book pages included (Soil weights,TCLP & SPLP). _____

Verify DOD QSM criteria and/or Project specific requirements. _____

Narrate any method deviations. (Blanks, LCS's etc.) _____

Sign & Date Manual integration **(Narrate as needed).** _____

Sample I.D's Truncated (**NARRATED**). YES ☐ Please list KAS # below :

_____

QA-044 – Revision 2 – 09/21/2010

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-502
Revision History
Cover Page
Page 1

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Prepared By: Michael Thomas Date: 07-24-00

Approved By:

Department Manager: [signature] Date: 6-23-06

Operations Manager: Deborah J. Nadeau Date: 6.23.06

QA Officer: Susie Dimond Date: 6-23-06

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
03	Changes to Sect. 5.5 & Figures 3 & 4 to reflect current spike solutions and concentrations Replaced cover page. Original cover page filed with SOP CA502-02	LAD	04/06	04/06
04	Added definitions, added waste information added LCSD, added SIM LCS/D, MS/D, updated Table 1, added use of narrow range pH paper. Minor changes throughout to reflect current practice.	LAD	09/07	09/07
05	Removed MS/MSD 14 day requirement. changed CLLE extraction time to 18 → 24 hours. Added information on determining initial sample volume. Added extracted Sample disposal. Removed all references to method 625.	LAD	09/08	09/08
06	Added to check pH after B/N CLLE extraction to ensure pH ≥ 11. If not add more NaOH and continue extracting. Added information for initial Volume determination. Added Reference to CA-109. Updated logbook example. Added if extract goes dry - re extract.	LAD	10/09	10/09
07	Sect. 5 - Removed baking and rinsing $NaSO_4$. Added 1,4-Dioxane to SIM surrogate Mix. Sect. 7 added acid to B/N SIM., removed to let separate for 10 minutes, minor edits throughout.	LAD	03/12	03/12

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Revision History (cont.):

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
08	Removed Sect. 7.1.9, determining the sample initial volume. Sect. 7.1.4 has this information. Figures 1 and 2 updated.	UAD	05/13	05/13

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-502-08**, titled **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-502-08**, titled **PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS**.

Recipient: _____ Date: _____

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe procedures utilized by Katahdin Analytical personnel in the preparation of all non-CLP aqueous samples for analysis of extractable semivolatile organic compounds.

The goal of this procedure is to ensure uniformity involving the preparation of samples for subsequent SVOA analysis by GC/MS. This SOP is applicable to EPA Methods 3510 (modified separatory funnel extraction) and 3520 (continuous liquid-liquid extraction), current revisions.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their department follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDS's for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, "Sample Disposal," current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware, disassembly of CLLEs after extraction, etc. should be disposed of in the "D" waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the "O" waste stream satellite accumulation area nearest the point of generation. Post-extraction aqueous samples are considered either N-Hi or N-Low waste and should be disposed of in the corresponding satellite waste accumulation area nearest the point of generation. Sodium sulfate used for sample drying should be disposed of in the soil with organics "I" waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

For aqueous samples extracted by CLLE, a one liter aliquot of sample is adjusted to pH $\leq$ 2 and extracted with methylene chloride using a continuous liquid-liquid extractor. The pH is then adjusted to pH $\geq$ 11 and the sample is extracted again with methylene chloride. A modified separatory funnel extraction may also be used. If this procedure is used, the sample aliquot is first adjusted to pH $\geq$ 11 and then to pH $\leq$ 2. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC/MS analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in the total ion current profiles (TICPs). Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure that clean glassware and apparatus are used and pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Brand names and catalog numbers are included for illustration purposes only.

4.1 Continuous liquid-liquid extractors - including body, 500 mL round bottom flask and Alhin condensers and equipped with Teflon or glass connecting joints requiring no lubrication (Hershberg-Wolf Extractor, Ace Glass Company, Vineland, NJ, P/N 6841-10 or equivalent).

4.2 Glass powder funnels.

4.3 Fluted filter paper, 18.5cm diameter.

4.4 Concentrator tube - Kuderna-Danish, 10 mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test.

4.5 Evaporation flask - Kuderna-Danish, 500 mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with neck clips.

4.6 Snyder column - Kuderna-Danish, three- or four-ball macro (Kontes K-503000-0121 or equivalent).

4.7 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.

4.8 Vials - Glass, 1.8 mL capacity, with polytetrafluoroethylene (PTFE)-lined screw top and 12 mL with Teflon-lined caps.

4.9 2 L separatory funnel, equipped with Teflon stopper and stopcock; Nalgene Teflon FEP separatory funnels may also be used.

4.10 Organic Free Boiling Chips - approximately 10/40 mesh, Teflon or silicon carbide (or equivalent). Cleaned by Soxhlet for 18 hours.

4.11 Water bath - heated, with concentric ring cover, capable of temperature control (± 20°C). The bath should be used in a hood.

4.12 Nitrogen evaporation apparatus.

4.13 Wide range pH test strips, pH 0-14, Whatman CF Type.

4.14 Glass rods for stirring samples.

4.15 Amber bottles or other appropriate containers for collection of extracts from separatory funnel extraction.

4.16 5 ¾" Pasteur pipets.

4.17 Narrow range pH test strips, pH 0 to 2.5 pH, EMD ColorpHast or equivalent.

4.18 Narrow range pH test strips, pH 11 to 13 pH, EMD ColorpHast or equivalent.

5.0 REAGENTS

All reagent and solvent lots must be checked for possible contamination. Refer to the current version of Katahdin SOP CA-105, Reagent and Solvent Handling, for further details. The extraction staff is responsible for submitting samples to the GC or GC/MS sections for appropriate analysis. All information concerning preparation of the reagent/solvent lot sample will be recorded in the Organic Extraction Log (Figure 1) and acceptance or rejection of these lots must be recorded in the solvent/reagent lot check logbook (Figure 2). All reagents and solvents must be free (<PQL) of any target compounds.

5.1 Laboratory Reagent Grade Water - defined as water in which an interferent is not observed at or above the PQL of each parameter of interest. Deionized water filtered through activated charcoal.

5.2 Sodium sulfate - (ACS reagent grade) granular, anhydrous, certified by the manufacturer/vendor as purified.

5.3 Sulfuric acid solution (1:1 H_2SO_4 : H_2O) – Prepared in an icebath by slowly adding a volume of concentrated H_2SO_4 to an equivalent volume of reagent water and swirl gently to mix. Caution should be taken when adding the acid to the water as the reaction is highly exothermic.

5.4 Acetone, methanol, methylene chloride - pesticide residue analysis grade or equivalent, evaluated prior to use by concentration of 200 mLs to 1.0 mL followed by GC and/or GC/MS analysis.

5.5 Standard Preparation - For all standard preparations, see current revision of the following Katahdin Analytical SOPs:

- "Standards Preparation, Documentation and Traceability", (CA-106, current revision)

- "Balance Calibration," (CA-102, current revision)

5.5.1. Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5.2 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	4.0 ug/mL
1,4-Dioxane-d8	20 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5.3 SVOA Matrix Spike/Lab Control Samples Spiking Solution - the matrix spike/LCS solution consists of the compounds listed in Figure 3.
Prepare a spiking solution that contains each of the base/neutral compounds listed in Figure 3 at 50 ug/mL in methanol and the acid compounds at 100 ug/mL in methanol. Matrix spike/LCS standards are stored in the freezer (-10°C to -20°C) located in the storage area.

5.5.4 Base/Neutral/Acid (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 2 ug/mL for base/neutrals and 4.0 ug/mL for acids. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL of methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months or sooner if comparison with quality control check samples indicates a problem.

5.5.5 Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution – Prepare a spiking solution in methanol that contains the compounds listed in Figure 4 at concentrations of 100 ug/ml. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Continuous liquid-liquid (Method 3520) and/or separatory funnel (Method 3510) extractions for semivolatiles must be started within seven days of date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific. If sampling date is unknown, the hold time is counted from one day prior to date received.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- Sample pH if applicable
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Any sample cleanup preformed
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

- Prep batch start time and end time
- CLLE start time and end time
- Lot number of the vials the concentrated extracts are stored in.

Follow the proper procedures for maintaining Internal Chain of Custodies for samples when removing and replacing samples in storage locations. This procedure is described in KAS SOP SD-902, "Sample Receipt and Internal Control", current revision.

7.1 CONTINUOUS LIQUID-LIQUID EXTRACTION (Method 3520)

7.1.1 Set up the CLLE apparatus. All glassware should be pre-rinsed three times with methylene chloride in order to eliminate any contamination factors.

7.1.2 Add approximately 500 - 600 mL of methylene chloride to the CLLE body. Label each flask with the following: sample number (or QC identification number), analyte (SVOA), extraction method (CLLE), and extraction date.

7.1.3 A method blank and a laboratory control sample (LCS) must be prepared for each daily extraction batch of twenty samples or fewer (if a work order consists of more than twenty samples, a new batch must be started on a separate page with its own method blank and LCS). To prepare method blank and LCS, add 1 L reagent water to a CLLE body. Be sure that no water leaks into the round bottom flask. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. This blank and LCS are carried through the entire extraction and analytical procedure.

7.1.4 Measure the initial volume by comparing the meniscus of the sample with the reference bottle of the same bottle type. Please refer to SOP CA-108, "Basic Laboratory Technique", for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.1.5 Transfer the sample to a CLLE body slowly, being sure that no water leaks into the round bottom flask.

7.1.6 If the batch requires a MS/MSD, transfer two 1 L portions of the sample selected/designated for MS/MSD to CLLE bodies for preparation of a matrix spike/matrix spike duplicate if required. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis. If extra MS/MSD aliquots of sample are unavailable a laboratory control sample duplicate (LCSD) may be substituted.

7.1.7 Check the pH of each sample with wide range pH paper by removing a couple of sample drops with a clean disposable pipet or on the tip of a stirring rod. Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to $\leq$ pH 2 with 1:1 H_2SO_4 after addition of surrogates and spikes and prior to attaching Allihn condensers (Step 7.1.11). Stir with a glass stirring rod and check pH by tapping the glassrod onto wide range pH paper. The pH must be ≤ 2. If the pH test strip does not clearly indicate the pH is less than 2, narrow range pH paper must be used.

7.1.8 For each sample, rinse the original sample container with approximately 30 mL of methylene chloride. Add this rinse to the CLLE body.

7.1.9 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL base/neutral and acid (SVOA) surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use.

7.1.9.1 If the request is for SVOA, use the SVOA Surrogate Solution (sect. 5.5.1).

7.1.9.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5.2).

7.1.9.3 If the request is for SIM-SVOA, use the SIM surrogate solution as well as SVOA surrogate solution. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.1.10 To LCS/LCSD and MS/MSD add 1.0 mL base/neutral and acid (SVOA) matrix spike/LCS spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in extraction logbook. Thoroughly rinse syringe with methanol before and after each use.

7.1.10.1 If the request is for SVOA - add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3).

7.1.10.2 If the request is for SIM - add 1.0 mL of SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3) and add 1.0 mL of Base/Neutral/Acid (SIM) Matrix Spike/ Lab Control Sample Spike Solution (sect 5.5.4).

7.1.10.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution as well as the SVOA spiking solution -add 1.0 mL of

SVOA Matrix Spike/Lab Control Samples Spiking Solution (sect 5.5.3) and add 1.0 mL of Base/Neutral and Acid (SVOA) Appendix IX Lab Control Sample / Matrix Spike Spiking Solution (sect 5.5.5).

7.1.11 Attach cooling water Allihn condensers, after first rinsing each 45/50 joint with methylene chloride. Turn on the heating mantles and allow the samples to extract for 18 to 24 hours. Turn off the mantles and let samples cool.

7.1.12 Detach condensers and verify that the pH is still $\leq$ 2 in the same manner mentioned in 7.1.6. If the pH has changed, more acid should be added to make the pH $\leq$ 2 and the sample extracted for several more hours.

7.1.13 Upon completion of acid extraction, allow the sample to cool. Detach condensers and add enough 10N NaOH to adjust the pH to $\geq$ 11 with stirring. Use glass stirring rods to stir and check the pH of each sample in the same manner mentioned in 7.1.6.

7.1.14 Re-attach Allihn condensers, turn on heating mantles, and allow samples to extract for 18 to 24 hours. Turn off mantles and allow samples to cool.

7.1.15 Detach condensers and verify that the pH is still $\geq$ 11 in the same manner mentioned in 7.1.6. If the pH has changed, more NaOH should be added to make the pH $\geq$ 11 and the sample extracted for several more hours.

7.1.16 Once samples are cool to the touch, the CLLE apparatus can be disassembled. The round bottom flask is removed, covered foil and placed in the interim extract refrigerator. The remaining sample in the CLLE body is poured in the "N-Hi" satellite.

7.1.17 Proceed to Step 7.3 for sample extract concentration procedures.

7.2 SEPARATORY FUNNEL EXTRACTION (Modified Method 3510)

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interference, then samples should be extracted by continuous liquid-liquid extraction (CLLE).

7.2.1 Rinse all glassware, including teflon separatory funnels, three times with methylene chloride prior to use.

7.2.2 Label 2 L separatory funnels and amber collection bottles clearly. Each label should include: sample number (or QC indicator number), analyte (SVOA), matrix (Aq), extraction date.

7.2.3 A method blank and a laboratory control sample (LCS) must be prepared for every 20 samples or with each extraction batch, whichever is more frequent. To prepare method blank and LCS, add 1 L reagent water to a separatory funnel. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. This blank and LCS are carried through the entire extraction and analytical procedure.

7.2.4 Measure the initial volume by comparing the meniscus of the sample with the reference bottle of the same bottle type. Please refer to SOP CA-108, "Basic Laboratory Technique", for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook.

7.2.5 If the batch requires a MS/MSD, transfer two 1 L portions of the sample selected/designated for MS/MSD to separatory funnels for preparation of a matrix spike/matrix spike duplicate if required. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis. If extra MS/MSD aliquots of sample are unavailable, a laboratory control sample duplicate (LCSD) may be substituted.

7.2.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL base/neutral and acid (SVOA) surrogate spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with acetone before and after each use.

7.2.6.1 If the request is for SVOA, use the SVOA Surrogate Solution.

7.2.6.2 If the request is for SIM, use the SIM Surrogate Solution.

7.2.6.3 If the request is for SIM-SVOA, use the SIM surrogate solution as well as SVOA surrogate solution. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.2.7 To LCS/LCSD and MS/MSD add 1.0 mL base/neutral and acid (SVOA) matrix spike/LCS spiking solution using a 1.0 mL or 2.5 mL gas tight syringe. Record matrix spike/LCS spiking solution volume and identification code in the extraction logbook. Thoroughly rinse syringe with methanol before and after each use.

7.2.7.1 If the request is for SVOA, use the SVOA Spiking Solution.

7.2.7.2 If the request is for SIM, use the SIM Spiking solution.

7.2.7.3 If the request is for SVOA Appendix IX, use the SVOA Appendix IX Spiking solution as well as the SVOA spiking solution

7.2.8 For each sample, rinse the original sample container with 60 mL of methylene chloride. Add this rinse to the separatory funnel.

7.2.9 Adjust the pH of the samples (including method blank, LCS/LCSD, and MS/MSD) to pH $\geq$ 11 with 10N NaOH after addition of surrogates and spikes. Stir with a glass stirring rod and check pH by tapping the glass stirring rod onto wide range pH paper. The pH must be $\geq$ 11. If the pH test strip does not clearly indicate the pH is greater than 11, narrow range pH paper must be used.

7.2.10 Add 60 mL of methylene chloride directly to the method blank and LCS/LCSD separatory funnels.

7.2.11 Extract the samples by shaking the funnel for two minutes, venting often, but gently, in a hood to release pressure. A mechanical shaker may be used, where samples are shaken for 3 minutes. Following each shake, allow phases to separate. Drain the methylene chloride layer into an amber collection bottle.

7.2.12 If an emulsion forms, mechanical techniques must be employed to achieve maximum separation. Such means include swirling, centrifugation, and draining through a small separatory funnel. In certain instances, transferring the entire sample into a continuous liquid-liquid extractor (CLLE) may be the only alternative. If any such techniques are used, they must be noted in the extractions logbook, and the batch transferred to a CLLE batch with its own batch ID.

7.2.13 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time (see 7.2.12 – 7.2.13). Collect the methylene chloride layer in the same amber collection bottle.

7.2.14 Repeat the extraction for a third time as described in 7.2.14.

7.2.15 Following the third shake, using a glass stirring rod, check the pH to ensure that it has remained at $\geq$ 11. If the pH has changed back to neutral range, it must be readjusted to $\geq$ 11 and the sample must be extracted at least one more time, adding the methylene chloride to the same amber bottle, that was previously used. If the pH has remained at a value $\geq$ 11, the pH is then adjusted to $\leq$ 2 with 1:1 H_2SO_4. Add enough 1:1 H_2SO_4 to adjust the pH to $\leq$ 2 with stirring. Use glass stirring rods to stir.

7.2.16 Add 60 mL methylene chloride and extract the samples three times in the same manner described in 7.2.11 – 7.2.13. Collect the methylene chloride layer in the same amber collection bottle used to collect the acid fraction.

7.2.17 Sample waste should be poured into the "n-lo" satellite.

7.2.18 Proceed to Section 7.3 for extract concentration procedures.

7.3 CONCENTRATING THE EXTRACTS

7.3.1 For Methods 3510 and 3520, the combined fractions are concentrated to a final volume of 1.0 mL.

7.3.2 Rinse the K-D glassware (flask, concentration tube, and snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride. Add two boiling chips to the K-D prior to final rinse. Also rinse the assembled funnels, filter paper, and granular sodium sulfate used for drying the extracts.

7.3.3 Transfer the methylene chloride extract to a K-D concentrator setup through a short stem funnel filled with 1-2 inches of sodium sulfate in filter paper. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain

7.3.4 Transfer the label from the collection bottle or round bottom flask (for CLLE) to a K-D. Remove the funnel and attach a 3- or 4-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.3.5 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.3.6 Reduce the methylene chloride extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook.

7.3.7 Reduce each extract to slightly less than 1 mL and then, using a 5 ¾" pasteur pipet, transfer the final extract and label to a 1.8 mL vial with PTFE-lined cap.

7.3.8 If at any time during the concentration process the concentrator tube goes dry, reextraction must occur immediately.

7.3.9 Transfer all of the extract to a 1.8 mL screw cap vial. Using methylene chloride, adjust the final volume of each extract to 1 mL by comparison to an appropriate reference vial.

Store in refrigerator until GC/MS analysis.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of semivolatiles for quality control acceptance criteria.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOP.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Methods 3510 and 3520, current revisions.

LIST OF TABLES AND FIGURES

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-502-08	METHOD 3510, current revision
Apparatus/Materials	1) 250 mL amber bottle or flask 2) 1.0 mL syringe 3) short stem funnels	1) 250 mL Erlenmeyer flask 2) 5.0 mL syringe 3) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) extract collection in amber bottle or Erlenmeyer flask 2) Add surrogate/spike to sample in CLLE 3) Extract for 3 minutes on mechanical shaker 4) extract three times at pH $\geq$ 11, then extract three times at pH $\leq$ 2. 5) extract dried using Na_2SO_4 in short stem funnels 6) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 7) water bath temp 75-85 deg C 8) no apparatus height specification for concentration on water bath 9) sample removed from water bath when volume reaches ~6 mL 10) N bath temp no higher than 39 deg C	1) extract collection in Erlenmeyer flask 2) Add surrogate/spike directly to sample bottle 3) Extract by shaking vigorously for 1 - 2 minutes with periodic venting 4) extract three times at pH $\leq$ 2, then extract three times at pH $\geq$ 11. 5) extract dried using Na_2SO_4 in drying columns 6) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer 7) water bath temp 15-20 deg C above solvent boiling temp 8) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min 9) sample removed from water bath when volume reaches 1 mL 10) N bath temp 35 deg C
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

TABLE 1, continued

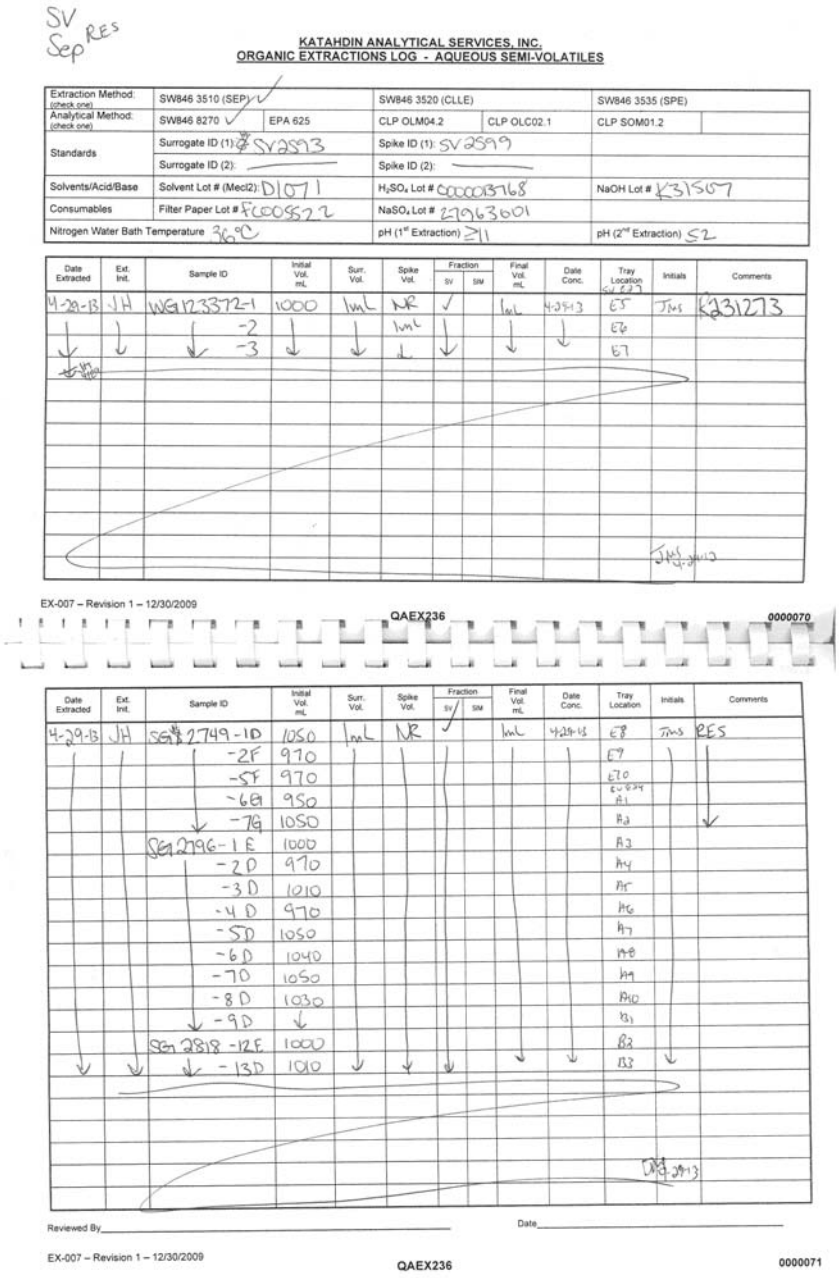
SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-502-08	METHOD 3520, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) Add surrogate/spike to sample in CLLE 2) Add approximately 500 - 600 mL of methylene chloride to the CLLE body 3) CLLE for 22 $\pm$ 2 hours 4) Extract dried using Na_2SO_4 in short stem funnels 5) Rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride then rinse the sodium sulfate with ~ 15 mLs of methylene chloride to complete a quantitative transfer 6) water bath temp 75-85 deg C 7) no apparatus height specification for concentration on water bath 8) sample removed from water bath when volume reaches ~6 mL 9) N bath temp no higher than 39 deg C	1) Add surrogate/spike directly to sample bottle 2) Add 300 - 500 mL of methylene chloride to the distilling flask of the extractor 3) CLLE for 18 - 24 hours 4) Extract dried using Na_2SO_4 in drying columns 5) Rinse the Erlenmeyer flask, which contained the solvent extract, with 20 - 30 mL of methylene chloride to complete the quantitative transfer 6) water bath temp 15-20 deg C above solvent boiling temp 7) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-20 min 8) sample removed from water bath when volume reaches 1 mL 9) N bath temp 35 deg C
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 1

EXAMPLE OF SEMIVOLATILES LOGBOOK PAGE



SV Sep RES

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - AQUEOUS SEMI-VOLATILES

Extraction Method: (check one)	SW846 3510 (SEP) ✓		SW846 3520 (CLLE)		SW846 3535 (SPE)	
Analytical Method: (check one)	SW846 8270 ✓	EPA 625	CLP OLM04.2	CLP OLC02.1	CLP SOM01.2	
Standards	Surrogate ID (1): SV2893		Spike ID (1): SV2899			
	Surrogate ID (2):		Spike ID (2):			
Solvents/Acid/Base	Solvent Lot # (MeCl2): DI071		H_2SO_4 Lot # 0000013768		NaOH Lot # K31507	
Consumables	Filter Paper Lot # FC005522		$NaSO_4$ Lot # 279963001			
Nitrogen Water Bath Temperature 36°C			pH (1st Extraction) ≥11		pH (2nd Extraction) ≤2	

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction SV	Fraction SIM	Final Vol. mL	Date Conc.	Tray Location	Initials	Comments
4-29-13	JH	WG123372-1	1000	1mL	NR	✓		1mL	4-29-13	E5	JMS	K231273
		-2			1mL					E6		
		-3								E7		

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Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction SV	Fraction SIM	Final Vol. mL	Date Conc.	Tray Location	Initials	Comments
4-29-13	JH	SG#2749-1D	1050	1mL	NR	✓		1mL	4-29-13	E8	JMS	RES
		-2F	970							E9		
		-5F	970							E10		
		-6G	950							A1		
		-7G	1050							A2		
		SG2796-1E	1000							A3		
		-2D	970							A4		
		-3D	1010							A5		
		-4D	970							A6		
		-5D	1050							A7		
		-6D	1040							A8		
		-7D	1050							A9		
		-8D	1030							A10		
		-9D								A11		
		SG2818-12E	1000							A12		
		-13D	1010							A13		

Reviewed By_____ Date_____

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TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 2

SOLVENT/REAGENT LOT CHECK LOGBOOK

GCMS

SOLVENT LOT CHECK

SOLVENT: Acetone

LOT#: DG818

DATE RECEIVED:

DATE CONCENTRATED: 4-8-13

CONCENTRATED BY: KF

PREP METHOD: 300mL → 1mL

TRAY LOCATION: SLC2 (A9)

ANALYZED BY: JCh

PASS/FAIL: Pass

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 3

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis(2-Ethylhexyl)adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl phthalate
3,3'-Dichlorobenzidine	Diethyl adipate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR EXTRACTABLE SEMIVOLATILE ANALYSIS

FIGURE 4

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

Prepared By: Mike Thomas Date: 09/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: J C Bouta Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10.24.00

General Manager: Dennis F. Hughes Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	10.24.00	10/24/00
02	Addition of compounds to Figure 2.	DN	3.28.02	3.28.02
03	Definitions added to section 1.1. Wording was added or changed to clarify sections 4, 5, 6, 7, 8 + 9. Minor changes throughout. New figures.	HRC	11.08.04	11.08.04
04	Updated Sect. 5.0 with current spike solutions prep. Removed section on medium level soil extraction. Replaced figure 3 and 4 with current LCS/MS spike components. Minor corrections to sect. 1.3, 4.24, 6.0 and 7.12. Updated logbook	LAD	04/06	04/06
05	Many changes made throughout, including but not limited to, waste information, updated spikes and surrogates, added SIM LCS/D and MS/D information, updated Table 1. Please refer to the QAM/SOP change form filed w/ SOP in QA for a detailed list of changes.	LAD	09/07	09/07

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Updated logbook example. Added adipate compounds to fig. 2. Added necessity of recording lot numbers of consumables in logbook. Added to record the temperature of the nitrogen evaporation water bath.	LAD	07/08	07/08
07	Added requirement to add spike before Na_2SO_4. Changed N_2 waterbath temperature from < 39°C to < 30°C. Removed respirator reference. Added KAES manual. Added KAS SOP CA-103 reference for additional subsampling information.	LAD	02/09	02/09
08	Removed targeting sample weights. Added KAT. SOP SD-902 reference. Updated logbook page example. Added GPC cleanup is required for all samples. Removed decanting samples	LAD	08/10	08/10
09	Minor modifications made to sections 5 & 7 to reflect current practices. Updated Section 9, to include LOD/LOQ requirements. Changed 7.6 & 7.7 to add surrogate and spikes after sodium sulfate is added. Updated references in Section 10.	LAD	04/12	04/12

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

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TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures and requirements for the preparation of solid samples for analysis of extractable semivolatile organic compounds. This SOP is specifically applicable to EPA Method 3550B in accordance with SW-846 Method 8270, current revision.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for semivolatile analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training”.

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for semivolatile analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab

notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and methanol are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples and sodium sulfate waste should be disposed of in the soil with organics “I” waste stream satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

2.0 SUMMARY OF METHOD

An approximate, greater than 30 gram portion of sediment/soil is mixed with anhydrous powdered sodium sulfate and extracted with 1:1 methylene chloride/acetone (v/v) using an ultrasonic probe. The methylene chloride extract is dried and concentrated to a volume of 1.0 mL.

3.0 INTERFERENCES

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause method interferences such as discrete artifacts and/or elevated baselines in the total ion current profiles (TICPs). All of these materials routinely must be demonstrated to be free from interferences under the conditions of the analysis by running laboratory method blanks. Interferences caused by phthalate esters can pose a major problem in semivolatile organics analysis because many phthalates are also target analytes. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis. Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with methylene chloride. Brand names and catalog numbers are included below for illustration purposes only.

4.1 Syringe - gas tight, 1.0 mL, solvent rinsed between each use.

4.2 Sonicator ultrasonic processor XL – Misonix (or equivalent) equipped with dual titanium 3/4" horn extenders for extracting two samples at a time.

4.3 Powder funnels, 100 mm diameter, 35 mm stem

4.4 Kuderna-Danish (KD) apparatus - Concentrator tube - 10 mL
Evaporative flask - 500 mL
Snyder column - 3-ball macro

4.5 Filter paper, 7.0 cm, Whatman #4

4.6 Vacuum filtration flask - 500 mL Erlenmeyer

4.7 Buchner funnel, porcelain, Coors® with 85 mm plate diameter (or equivalent)

4.8 Beakers - 400 mL

4.9 Boiling chips - approximately 12 mesh, silicon carbide (carborundum or equivalent). Soxhlet extract overnight in methylene chloride.

4.10 Water bath - eight position concentric ring bath, or equivalent, equipped with a calibrated thermometer. The bath should be used in a hood.

4.11 Balance - capable of accurately weighing ± 0.01 g.

4.12 Vials and caps – 1.8 mL with PTFE/silicone septa and 12 mL with Teflon-lined caps for extracts designated for GPC cleanup.

4.13 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.14 Pasteur pipets - disposable, 5 ¾ ".

4.15 Nitrogen evaporation apparatus.

4.16 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.17 Gel Permeation Chromatograph (GPC) - J2 Scientific AccuPrep MPS™ with internal UV detection

5.0 REAGENTS

5.1 Sodium Sulfate - anhydrous powdered and granular crystals, reagent grade, certified by the manufacturer/vendor as purified heating to 400°C prior to receipt by the laboratory. (Jost Chemical anhydrous powder, catalog #2797 or equivalent, and Jost Chemical granular crystals, catalog #2796 or equivalent).

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

5.2 Methylene chloride, methanol, and acetone - pesticide residue analysis grade or equivalent. Methylene chloride and acetone are evaluated by lot prior to use by concentration of approximately 200 mL to 1.0 mL followed by GC/MS analysis. The lot numbers of all solvents used during an extraction must be recorded in the extraction logbook.

5.3 Organic-free sand, purified by baking at 400 °C. Method blanks serve as checks on the baked sand.

5.4 Base/Neutral and Acid (SVOA) Surrogate Spiking Solution - Surrogate standards are added to all samples and calibration solutions. Prepare a surrogate standard spiking solution that contains the following compounds at the indicated concentrations in acetone.

Compound	Conc.
phenol-$_{d6}$	100 ug/mL
2,4,6-tribromophenol	100 ug/mL
2-fluorophenol	100 ug/mL
nitrobenzene-$_{d5}$	50 ug/mL
p-terphenyl-$_{d14}$	50 ug/mL
2-fluorobiphenyl	50 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.5 SIM Surrogate Spiking Solution- Surrogate Standards are added to all samples and calibration solutions. Prepare a surrogate solution that contains the following compounds at a concentration of 2 ug/mL in acetone.

Compound	Conc. ug/mL
Fluorene-d10	2.0 ug/mL
2-Methylnaphthalene-d10	2.0 ug/mL
Pyrene-d10.	2.0 ug/mL
2,4-Dibromophenol	4.0 ug/mL
1,4-Dioxane-d8	2.0 ug/mL

Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem. If reanalysis of a method blank still indicates surrogates out of criteria, a new surrogate solution must be used.

5.6 Base/Neutral and Acid (SVOA) Matrix Spike/Lab Control Sample Spiking Solution - Prepare a spiking solution in methanol that contains the compounds listed in Figure 2

at a concentration of 50 ug/mL for base/neutrals and 100 ug/mL for acids. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

5.7 Base/Neutral and Acid (SVOA APPENDIX IX) Matrix Spike/Lab Control Sample Spiking Solution. Prepare a spiking solution in methanol that contains the compounds listed in Figure 3 at a concentration of 100 µg/mL for each compound. Store the spiking solution at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem

5.8 Base/Neutral and Acid (SIM) Matrix Spike/ Lab Control Sample Spike Solution for SIM-SVOA. Prepare a spiking solution in methanol that contains the compounds listed in Figure 2 at a concentration of 2.0 ug/mL for base/neutral and 4.0 ug/mL for acid. Take out 1.0 mL of Base/Neutral and Acid Matrix Spike/Lab Control Spiking Solution for SVOA and dilute it to 25.0 mL in methanol. Store the solution Spiking at -10°C to -20°C in Teflon-sealed containers. These solutions must be replaced after six months, or sooner if comparison with quality control check samples indicates a problem.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3550 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- pH if applicable

- Sonicator horns tuned
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

All solid samples should be cleaned using gel permeation chromatography (GPC) to reduce matrix interferences.

The organic department manager should be consulted to determine if a particular sample should be subjected to further cleanup procedures; the decision should consider sample history, sample appearance, and project/client needs.

Samples need to be "swiped" out when removing and "swiped" in when replacing samples in storage locations to maintain the internal chain of custody. Refer to Katahdin SOP SD-902, Sample Receipt and Internal Control, current revision, for the proper procedure for removal and return samples. Fill out the sample preparation/extraction log with the necessary information before starting the extraction. Prerinse all glassware three times with methylene chloride.

7.1 Do not decant any water layer on a sediment sample. Mix with a stainless steel spatula to ensure homogeneity of the sample. If the sample container is full to the extent that stirring the sample is impractical, try to remove the "best representative" aliquot from the jar based on color, particle size, moisture, etc. Remove any foreign objects such as sticks, leaves, and rocks, and note actions taken in the appropriate extraction logbook. Please refer to the current revision of Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique ", for more detailed guidance on subsampling to ensure reproducibility.

7.2 The following steps should be performed rapidly to avoid loss of the more volatile extractable. Weigh out an approximate, greater than 30g portion of sample into a labeled 400-mL beaker. Record sample weight to the nearest 0.01 g in appropriate extraction logbook. Add between 30 g and 60 g of anhydrous powdered sodium sulfate as required for producing a "free-flowing" mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil.

7.3 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare a method blank, weigh out one, greater than 30 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.6 and 7.7

for spike and surrogate addition instructions. Add 60 g sodium sulfate and mix well. Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.

7.4 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one 30 g portion of purified sand in a labeled 400 mL beaker. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 30 g sodium sulfate and mix well. If combined SIM-SVOA analysis is requested, a separate LCS must be prepared for each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.5 A matrix spike/matrix spike duplicate (MS/MSD) set must be prepared for every 20 samples. To prepare MS/MSD, weigh out two approximate, greater than 30 g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Refer to sections 7.6 and 7.7 for spike and surrogate addition instructions. Add 30 - 60 g sodium sulfate to each to produce a free-flowing mixture, and mix well. If combined SIM-SVOA analysis is requested, a separate MS/MSD must be prepared for each analysis.

7.6 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL of the appropriate base/neutral and acid surrogate spiking solution listed below using the pre-rinsed 1.0 mL gas tight syringe. The surrogate spike should be added **after** the addition of the sodium sulfate. Record surrogate spike volume and identification code in extraction logbook. Thoroughly rinse syringe with solvent prior to using it for another spiking solution.

7.6.1 If the request is for SVOA or SVOA Appendix IX, use the SVOA surrogate solution (sect. 5.4).

7.6.2 If the request is for SIM, use the SIM surrogate solution (sect. 5.5).

7.6.3 If the request is for SIM-SVOA, use both the SIM and SVOA surrogate solutions. NOTE: Separate LCS/LCSD and/or MS/MSD are needed for each analysis and should be spiked with the appropriate surrogate.

7.7 To the LCS/LCSD and the MS/MSD add 1.0 mL of the appropriate base/neutral and acid (SVOA) matrix spike/LCS spiking solution listed below using a 1.0 mL gas tight syringe. The LCS/MS spike should be added **after** the addition of the sodium sulfate. Record the matrix spike/LCS spiking solution volume and identification

code in extraction logbook. Thoroughly rinse the syringe with solvent when spiking is completed.

7.7.1 If the request is for SVOA, add 1 mL of SVOA Spiking Solution (sect 5.6).

7.7.2 If the request is for SIM, add 1 mL SIM Spiking solution (sect 5.8).

7.7.3 If the request is for SVOA and SIM, add 1mL of SVOA Spiking Solution and 1 mL SIM Spiking solution (sect 5.6 and 5.8).

7.7.4 If the request is for SVOA Appendix IX, add 1mL of SVOA Spiking Solution and 1 mL of SVOA Appendix IX Spiking solution (sect 5.6 and 5.7).

7.8 To assure optimum operation and maximum energy output, the sonicators must be tuned daily prior to extracting samples. The following tuning procedure must be performed with the sonicator probes vibrating in air.

7.8.1 Turn OUTPUT CONTROL knob counter-clockwise to zero. This automatically switches the duty cycle to continuous mode.

7.8.2 Press and hold down the power switch to on.

7.8.3 Press and hold down the TUNE switch. Check if the counter is less or equal to 20%; otherwise, rotate the Tuning Knob (tuning button) clockwise until a reading of 20%or less is obtained.

7.8.4 Release the TUNE switch.

7.8.5 Turn OUTPUT CONTROL KNOB counter-clockwise to 50 and the power switch off.

7.8.6 Confirm that the sonicators were tuned by recording the date and/or percent in the extractions logbook.

7.9 Prior to extracting any samples, ensure that the sonicator probes are decontaminated by rinsing three times with a methylene chloride wash bottle. Collect the waste in a waste beaker. It may sometimes be necessary to wipe the upper part of each probe with a methylene chloride dampened KimWipe. Repeat this decontamination step between each sample on each probe.

7.10 To the mixed and spiked blank and LCS, add approximately 100 mL of the 1:1 methylene chloride/acetone (V/V) solution and proceed with steps 7.11 through 7.14. Record the lot numbers of the solvents in the extraction logbook.

7.11 It may be necessary at this time to stir the sample/sodium sulfate mixture with the spatula to loosen up the mixture prior to extracting. Rinse the spatula with methylene chloride and collect the rinsing into a correspondent beaker. Position the beaker in the ultrasonic cell disruptor so that the bottom surface of the tip of the 3/4 inch disruptor horn is about halfway below the surface of the solvent and above the sediment layer.

7.12 Sonicate for 3 minutes with the output control knob set at 10, and mode switch on "pulsed" and % duty cycle knob set at 50%. While the mixture is sonicating, one should be able to see all, or most of the material, moving in the beaker under the influence of the energized probes. If not, stir the mixture again.

7.13 Prepare a filter flask fitted with a Buchner funnel. The Buchner funnel should contain a 7.0 cm Whatman #4 filter. Prerinse the flask, funnel and filter with methylene chloride and discard rinsings into solvent waste container. Decant extract into the filter flask and Buchner funnel. A vacuum pump may be used to facilitate filtration or the extract may be gravity filtered. The lot number of the filter paper must be written ti the extraction logbook.

7.14 Repeat the extraction two more times (sec 7.11 – 7.14) using approximately 100 mL portions of 1:1 methylene chloride: acetone. Before each extraction, make certain that the sodium sulfate is still free-flowing and not a consolidated mass. As required, break up large lumps with the spatula. Decant the extraction solvent into the Buchner funnel after each sonication. On the final sonication, pour the entire sample contents into the Buchner funnel and rinse thoroughly with methylene chloride to complete the quantitative transfer of the extract. Use the vacuum pump to pull all the extract into the flask

CONCENTRATION OF LOW LEVEL EXTRACTS

7.15 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add two boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Place the assembled K-D's under the funnels. The lot number of the filter paper must be written in the extraction logbook.

7.16 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mls of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative

transfer. Rinse the sodium sulfate with ~ 15 mls of methylene chloride and allow to drain.

7.17 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures.

7.18 If samples are not to be GPC'd, follow Steps 7.19 through 7.24 to concentrate extracts to final volume of 1 mL. Otherwise proceed to GPC cleanup procedure as described in the current revision of Katahdin SOP CA-513.

7.19 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.20 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. **Do not allow the evaporator to go dry. If the sample extract does go dry, re-extraction must occur immediately.** Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride.

7.21 Reduce the extract in the concentrator tube to approximately 1 mL using the nitrogen blow-down apparatus. The bath temperature must be < 39°C. Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of methylene chloride. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the logbook also note any problems or extract losses, if they occur, in the extractions logbook.

7.22 When the apparent volume reaches slightly less than 1 mL, remove the concentrator tube and allow it to cool.

7.23 Complete the quantitative transfer of the extract to a 1.8 mL vial by using methylene chloride. Adjust the volume of the methylene chloride extract to 1.0 mL using the 1.8 mL reference vial for volume comparison.

7.24 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extraction logbook the box number and "tray location" of the individual extract vials.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples

Refer to the current revision of the applicable Katahdin SOP for analysis of Semivolatile Organics for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able

to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Detection (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3550C, USEPA SW-846, Third Edition, Update IV, February 2007.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Current Version.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

TABLE 1

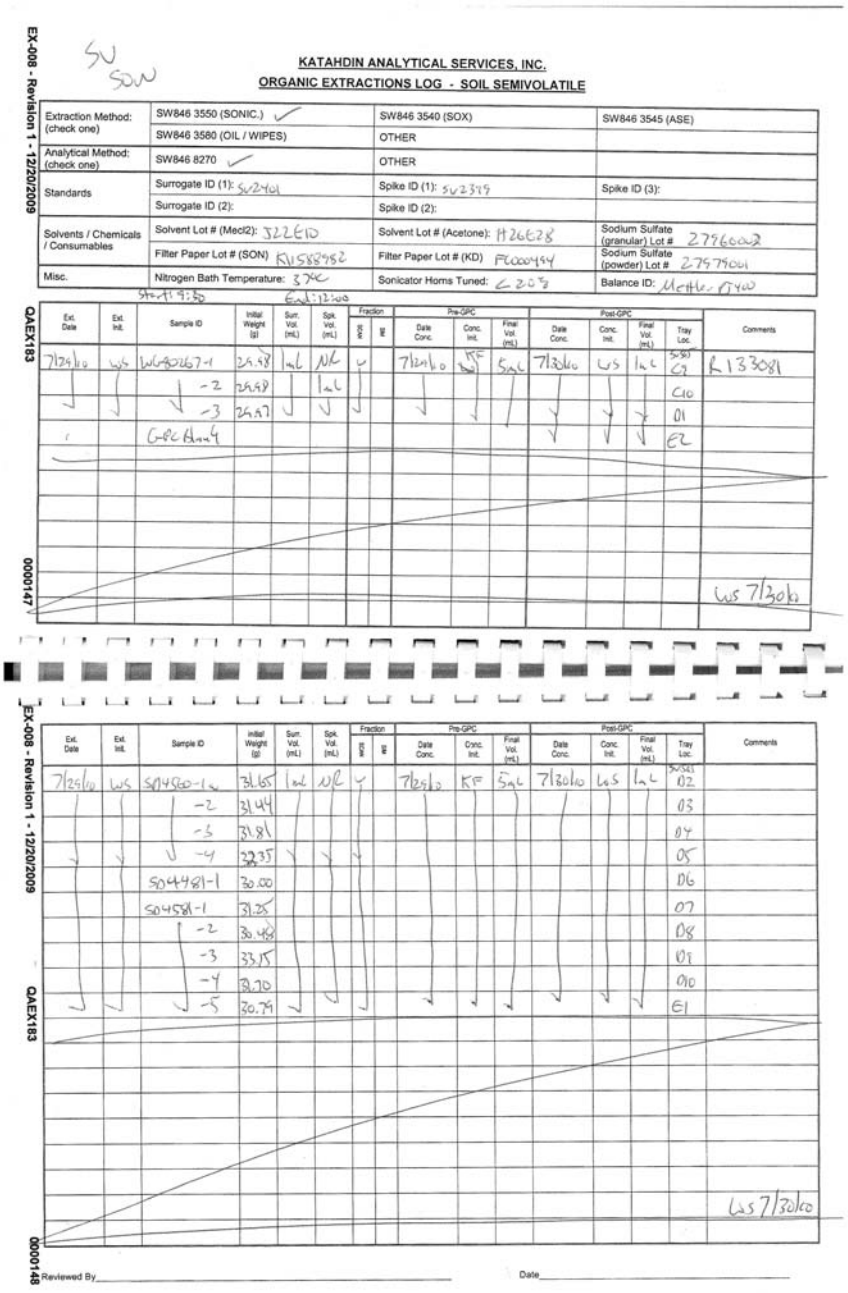
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-512-09	METHOD 3550, current revision
Apparatus/Materials	1) short stem funnels	1) drying columns
Reagents		
Sample preservation/ handling		
Procedures	1) extract dried using Na_2SO_4 in short stem funnels 2) place sonicator horns ½ way between the surface of the solvent and the sediment layer 3) no apparatus height specification for concentration on water bath 4) water bath at 75-85 deg C 5) sample removed from water bath when volume reaches ~6 mL	1) extract dried using Na_2SO_4 in drying columns 2) place sonicator horns ½ inch below the solvent surface but above sediment layer 3) partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4) water bath at 80-90 deg C 5) sample removed from water bath when volume reaches 1-2 mL
QC - Spikes	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - LCS	1) Acid surrogate and spike components at 100 ug/mL; base/neutral surrogate and spike components at 50 ug/mL	1) Acid surrogate and spike components at 200 ug/mL; base/neutral surrogate and spike components at 100 ug/mL
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL SEMIVOLATILE

Extraction Method: (check one)	SW846 3550 (SONIC.) ✓	SW846 3540 (SOX)	SW846 3545 (ASE)
	SW846 3580 (OIL / WIPES)	OTHER	
Analytical Method: (check one)	SW846 8270 ✓	OTHER	
Standards	Surrogate ID (1): SV2401	Spike ID (1): SV2379	Spike ID (3):
	Surrogate ID (2):	Spike ID (2):	
Solvents / Chemicals / Consumables	Solvent Lot # (MeCl2): J22E1D	Solvent Lot # (Acetone): H26E28	Sodium Sulfate (granular) Lot # 279600-2
	Filter Paper Lot # (SON) RU1588982	Filter Paper Lot # (KD) FC000494	Sodium Sulfate (powder) Lot # 275790-1
Misc.	Nitrogen Bath Temperature: 35°C	Sonicator Horns Tuned: ✓ 20%	Balance ID: Metler 6140

Start: 9:50 End: 12:40

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction ABN	Fraction BN	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
7/29/10	WS	WW502667-1	29.98	1mL	NR	✓		7/29/10	KF	5mL	7/30/10	WS	1mL	SV561 C9	R133081
↓	↓	-2	29.98	↓	1mL									C10	
↓	↓	-3	29.97	↓	↓	↓								D1	
		GPC Blank												E2	
															WS 7/30/10

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spk. Vol. (mL)	Fraction ABN	Fraction BN	Pre-GPC Date Conc.	Pre-GPC Conc. Init.	Pre-GPC Final Vol. (mL)	Post-GPC Date Conc.	Post-GPC Conc. Init.	Post-GPC Final Vol. (mL)	Tray Loc.	Comments
7/29/10	WS	504560-1	31.65	1mL	NR	✓		7/29/10	KF	5mL	7/30/10	WS	1mL	SV561 D2	
↓	↓	-2	31.44	↓	↓	↓								D3	
		-3	31.81											D4	
		-4	32.35											D5	
		504481-1	30.00											D6	
		504581-1	31.25											D7	
		-2	30.48											D8	
		-3	33.15											D9	
		-4	31.70											D10	
		-5	30.79											E1	
															WS 7/30/10

Reviewed By_____ Date_____

EX-008 - Revision 1 - 12/20/2009 QAEX183 0000147

EX-008 - Revision 1 - 12/20/2009 QAEX183 0000148

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 2

LCS/MATRIX SPIKE COMPONENT LIST

BASE/NEUTRALS	
1-Methylnaphthalene	Bis (2-chloroethoxy) methane
1,1-Biphenyl	Bis (2-chloroethyl) ether
1,2,4-Trichlorobenzene	Bis (2-Chloroisopropyl) ether)
1,2-Dichlorobenzene	Bis (2-ethylhexyl) adipate
1,3-Dichlorobenzene	Bis (2-ethylhexyl) phthalate
1,4-Dichlorobenzene	Butylbenzyl phthalate
1,4-Dioxane	Caprolactam
2,4-Dinitrotoluene	Carbazole
2,6-Dinitrotoluene	Chrysene
2-Chloronaphthalene	Dibenz (a, h) anthracene
2-Methylnaphthalene	Dibenzofuran
2-Nitroaniline	Diethyl adipate
3,3'-Dichlorobenzidine	Diethyl phthalate
3-Nitroaniline	Dimethyl phthalate
4-Bromophenylphenyl ether	Di-n-butylphthalate
4-Chloroaniline	Di-n-octyl phthalate
4-Chlorophenylphenyl ether	Fluoranthene
4-Nitroaniline	Fluorene
Acenaphthene	Hexachlorobenzene
Acenaphthylene	Hexachlorobutadiene
Acetophenone	Hexachlorocyclopentadiene
Aniline	Hexachloroethane
Anthracene	Indeno (1,2,3-cd) pyrene
Atrazine	Isophorone
Azobenzene	Naphthalene
Benzaldehyde	Nitrobenzene
Benzidine	N-Nitrosodimethylamine
Benzo (a) Anthracene	N-Nitroso-di-n-propylamine
Benzo (a) pyrene	N-Nitrosodiphenylamine
Benzo (b) fluoranthene	Phenanthrene
Benzo (ghi) perylene	p-toluidine
Benzo (k) fluoranthene	Pyrene
Benzyl alcohol	Pyridine

ACIDS		
2, 3, 4, 6-Tetrachlorophenol	2-Chlorophenol	Benzoic acid
2,4,5-Trichlorophenol	2-Methylphenol	Ethyl methanesulfonate
2,4,6-Trichlorophenol	2-Nitrophenol	Methyl methanesulfonate
2,4-Dichlorophenol	4,6-Dinitro-2-methylphenol	Pentachlorophenol
2,4-Dimethylphenol	4-Chloro-3-methylphenol	Phenol
2,4-Dinitrophenol	4-Methylphenol	
2,6-Dichlorophenol	4-Nitrophenol	

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SONICATION USING METHOD 3550 FOR SUBSEQUENT EXTRACTABLE SEMI-VOLATILES ANALYSIS**

FIGURE 3

APPENDIX IX LCS/MATRIX SPIKE COMPONENT LIST

1,2,4,5-Tetrachlorobenzene	Hexachloropropene
1,3,5-Trinitrobenzene	Isodrin
1,4-Naphthoquinone	Isosafrole
1-Chloronaphthalene	Kepone
1-Naphthylamine	m-Dinitrobenzene
2,4-D	Methapyrilene
2-Acetyl aminofluorene	Methyl parathion
2-Naphthylamine	n-Nitrosodiethylamine
2-Picoline	n-Nitrosodi-n-butylamine
3,3-Dimethylbenzidine	n-Nitrosomethylethylamine
3-Methylcholanthrene	n-Nitrosomorpholine
4-Aminobiphenyl	n-Nitrosopyrrolidine
4-Nitroquinoline-1-oxide	n-Nitrotrosopiperidine
5-Nitro-o-toluidine	O,O,O-Triethyl phosphorothioate
7,12-Dimethylbenz(a)anthracene	o-Toluidine
a,a-Dimethylphenethylamine	Parathion
Acetophenone	p-Dimethylaminoazobenzene
Aramite	Pentachlorobenzene
Chlorobenzilate	Pentachloronitriobenzene
Diallate	Phenacetin
Dibenz(a,j)acridine	Phorate
Dimethoate	p-Phenylenediamine
Dinoseb	Pronamide
Diphenylamine	Safrole
Disulfoton	Silvex (2,4,5-TP)
Famphur	Sulfotep
Hexachlorophene	Thionazin

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Jessica Spearh-Wildes

Review Date: 3-15-13

SOP Number: QA-512

SOP Title: Preparation of Sediment / Soil samples by Sonication using method 3550 for Semi-volatiles

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 3-15-13

QAO Signature: Leslie Dimmd **Date:** 031913

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Prepared By: GC/MS Department Date: 6/98

Approved By:

Group Supervisor: J. Halaj Date: 020101

Operations Manager: John C. Bentas Date: 1/31/01

QA Officer: Deborah J. Nadeau Date: 1.31.01

General Manager: Duncan F. Crafton Date: 2/01/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8270C Mod.	Format changes, added pollution prevention, added instrument and other calibration options. Other minor changes to sections 7, 8 & QA Table.	DN	1.31.01	1.31.01
02 8270C	Many changes in formatting. Some additions to sections 8 + Table 1 to comply with Navy.	DN	09.30.04	09.30.04
03 8270C	Sect. 7.2: Removed "K" Instrument; added "R" instrument. Added Pentafluorophenol surr. to Tables 3, 5 and Sect. 8.2. Removed all References to TIC's.	LAD	04/06	04/06
04 8270C	Sect. 8.2 - changed 5 to 4 and removed pentachlorophenol. Table 3 and 5 - removed pentachlorophenol. Changed linear regression correlation coefficient criteria. Added MI SOP reference. Added LCS exceedance criteria. Added ICV requirement and criteria. Added RT window procedure.	LAD	06/07	06/07
05 8270C	Added "G" instrument, Removed "X" instrument Edited section 7.5.1 - initial cal. table	LAD	02/08	02/08

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Section 5.3.2.3 - Added calibration Mix B. Section 7.5.1 - Edited to address diffent SIM compounds may need to be calibrated at different levels depending on the compound and project requirements.	LAD	04/09	04/09
07	Changes made for compliance with DoD QSM version 4.1	LAD	08/09	08/09
08	Updated Standard prep. Added compounds to Table 3 and 5. Updated references. Added DoD QSM QC requirements Table.	LAD	04/10	04/10
09	Sect. 7.4 - Added additional tune information. Sect. 7.6 - Added 100 ul minimum extract vol. & 1 ul IS is added for each 100 ul aliquot. Sect. 7.5.4 - Added RRT information. Sect. 9.0 - Added MDL, LOD and LOQ information. Table 4 - Added 1,4-Dioxane-d6 Surrogate	LAD	05/11	05/11
10	Sect. 7 - Changed sample volume from 1ul to 2ul. Sect. 8 - Added 10% rule for non-DoD clients. Sect. 9 - Added MDL, LOD and LOQ information. Sect. 10 - Added and updated references. Updated Figure 1. Added Addendum 1 - low level 1,4-Dioxane analysis	LAD	05/12	05/12
11	Sect. 1 and 7 - Removed Quickfacts reporting and added KIMS. Sect. 8 and Table 1 - Added the surrogate 1,4-Dioxane-d8. Throughout - Fixed typos and made minor changes.	LAD	03/13	03/13

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-213-11, titled "**ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**".

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-213-11, titled "**ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY METHOD 8270 – Modified for Selected Ion Monitoring (SIM)**".

Recipient: _____ Date: _____

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel to prepare and analyze water and soil sample extracts for semivolatile organics by EPA SW-846 Method 8270, current revision, modified for selected ion monitoring.

In order to maintain consistency in data quality, this SOP consolidates all aspects of the analyses in one working document, to be revised as necessary.

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.
STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS) : A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of semivolatile organic compounds by EPA Method 8270. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability,” current revision.

It is the responsibility of all Katahdin technical personnel involved in analysis of semivolatiles by Method 8270 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of

hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves, and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

After analysis, autosampler vials containing sample extracts in methylene chloride are returned to the SVOA hood, and the contents transferred to a labeled waste container. The contents of this container are disposed of in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

2.0 SUMMARY OF METHOD

The process involves the extraction of semivolatiles from a sample using an appropriate solvent followed by clean up steps (where applicable) and concentration of the extract (refer to Katahdin SOP CA-502, “Preparation of Aqueous Samples for Extractable Semivolatile Analyses”, SOP CA-512, “Preparation of Sediment/Soil Samples by Sonication Using Method 3550 for Subsequent Extractable Semi-Volatiles Analysis” and SOP CA-526, “Preparation of Sediment/Soil Samples by Soxhlet Extraction Using Method 3540 for Subsequent Extractable Semivolatile Analysis”). An aliquot of the final extract is injected into the gas chromatograph for compound separation by capillary column, followed by the electron impact mass spectrometer for identification and quantitation.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

3.0 INTERFERENCES

Interfering contamination may occur when a sample containing low concentrations of SVOCs is analyzed immediately after a sample containing high concentrations of SVOCs. Any samples that have suspected carryover must be reanalyzed.

4.0 APPARATUS AND MATERIALS

4.1 GC: Hewlett Packard 5890 and/or 6890

4.2 Mass Spectrometers (MS): HP5975, HP5973, HP5972 and/or HP5970

4.3 Helium: Carrier gas for routine applications. All carrier gas lines must be constructed from stainless steel or copper tubing; non-polytetrafluoroethylene (non-PTFE) thread sealant or flow controllers with rubber component are not to be used.

4.4 Autosamplers: HP 7673As

4.5 Hamilton syringes: 2.00 uL to 10 mL

4.6 Volumetric glassware: Grade A or equivalent

4.7 Columns: DB-5MS 30m, 0.25mm I.D., 25um film thickness, columns (J&W Scientific) or equivalent.

4.8 Acquisition System: The acquisition system must be interfaced to the MS and allow continuous acquisition of data throughout the duration of the chromatographic program. It must permit, at a minimum, the output of time vs. intensity (peak height or peak area). Hewlett Packard Chemstation or equivalent.

4.9 Data System: The Target software is used for processing data and generating forms.

5.0 REAGENTS

5.1 J.T. Baker Ultra Resi-Analyzed methylene chloride (or equivalent)

5.2 Purge and trap grade methanol

5.3 Standards: Stock standards and working standards are received and recorded in accordance with SOP CA-106 “Standard Preparation and Documentation”.

5.3.1 The expiration date for all standards is one year from date of opening the ampule. If the manufacturer's expiration date is before this one year date, the manufacturer's expiration must be followed. New standards must be opened if degradation is observed.

5.3.2 Secondary dilution standards

5.3.2.1 The standards are prepared on an as needed basis (or every 6 months) and stored in screw-cap amber bottles with Teflon liners in the BNA standards freezer between uses. Standards prepared from various stock solutions must always use the first expiration date of any of the solutions used for preparation.

5.3.2.2 Calibration Mix A – Prepare standards in methylene chloride containing the compounds listed in Table 3. The final concentration of each compound is 20 ug/mL.

5.3.2.3 Calibration Mix B - Some compounds must be calibrated at higher concentrations. For these compounds a secondary standard is prepared which will "boost" the concentration of these compounds in the initial calibration. The concentration of this standard is determined on a project to project basis.

5.3.2.4 Internal Standard Solution – Prepare standard in methylene chloride containing 1,4-dichlorobenzene-d4, naphthalene-d8, acenaphthene-d10, phenanthrene-d10, chrysene-d12, and perylene-d12 at a final concentration of 80 ug/mL.

5.3.2.5 DFTPP Solution – Prepare standard in methylene chloride containing DFTPP at a final concentration of 25 ug/mL.

5.3.2.6 Independent Calibration Verification (ICV) Standard – From a source independent of the calibration standards, prepare a standard in methylene chloride containing the compounds listed in Table 3. The final concentration of each compound is 2 ug/mL.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

All semivolatile sample extracts must be analyzed within forty days following the date of extraction.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

7.0 PROCEDURES

7.1 NAMING AND CODING CONVENTIONS FOR ANALYTICAL STANDARDS – Used in accordance with SOP CA-106 "Standard Preparation and Documentation".

7.2 COMPUTER (DATA SYSTEM) CONVENTIONS -

Conventions for all instruments are as follows:

Sub-Directory for data acquisition and storage: C:\HPCHEM\1\DATA
Tune file: DFTPP.U

Method files: LSPSIMXX.M (all samples and standards)
Where:
XX = the calibration number in chronological order
L = instrument ID (Each instrument is given a unique identifier)
DFTPP390.M (DFTPP tuning acquisition)

NOTE: All acquisition parameters must be identical for LSPSIMXX.M and DFTPP2. M.

Data Files: L_ _ _ _.D, where _ _ _ _ is a number in chronological order from 0001 to 9999 and L is the instrument ID (Each instrument is given a unique identifier). This file also contains the Quantitation output file.

Data Files for DFTPP: LD_ _ _.D, where _ _ _ is a number in chronological order from 001 to 999 and L is the instrument ID (Each instrument is given a unique identifier).

7.3 INSTRUMENT SPECIFIC PROCEDURES

It is the policy of the GC/MS group that all data be acquired in the batch mode. The following items must be checked prior to data acquisition in the batch mode:

- Ensure that the proper sequence and tune files are being used.
- Check the autosampler syringe (Is it clean? Does the plunger move freely? etc.), its alignment and make sure the solvent rinse vial is full. Ensure that the knurled nut holding the top of the syringe plunger is tight.
- Look at the batch to be analyzed and check the following:

Make sure that the data files are in numerical order with no duplication and that the method file is the same as that used for ICAL or Continuing Calibration analysis.

Bottle numbers match with the numbers on the autosampler tray.

After the batch has been deemed free of errors, start the batch by using the "Position and run" command under the SEQUENCE menu in MSTop.

7.4 INSTRUMENT TUNING - Prior to the analysis of any calibration standards, blanks or samples, the GC/MS system must be shown to meet the mass spectral key ion and ion abundance criteria for decafluorotriphenylphosphine (DFTPP) tabulated below. Pentachlorophenol, benzidine and DDT are also present in this standard.

DFTPP Key Ions and Ion Abundance Criteria	
Mass	**Criteria**
51	30.0-60.0 percent of mass 198
68	less than 2.0 percent of mass 69
69	present
70	less than 2.0 percent of mass 69
127	40.0 – 60.0 percent of mass 198
197	less than 1.0 percent of mass 198
198	base peak, 100 percent of mass 198
199	5.0-9.0 percent of mass 198
275	10.0-30.0 percent of mass 198
365	greater than 1.00 percent of mass 198
441	present, but less than mass 443
442	greater than 40.0 percent of mass 198
443	17.0-23.0 percent of mass 442

All ion abundances must be normalized to m/z 198, the nominal base peak.

The following are the GC/MS operating conditions for injection of DFTPP.

GC/MS Operating Conditions - DFTPP	
Initial column temperature hold	140°C for 3 minutes
Column temperature program	140-275°C at 15 degrees/minute
Final column temperature hold	275°C
Injection port temperature	280°C
Transfer line/source temperature	285°C
Injector - splitless, valve time	0.18 minutes
EPC	inlet B
Constant flow	ON
Constant flow pressure	10psi
Constant flow temperature	30°C
Vacuum comp.	ON
Run time	10-12 minutes
Scan start time	5.0 minutes
Sample volume	2.0 uL of 25 ng/uL DFTPP solution
Carrier gas	helium at @ 1.0 mL/minute
Mass range	35 to 500 amu
Number of A/D samples	4
GC Peak threshold	500 counts
Threshold	10 counts

Set up the run on the Enviroquant system using "Edit Sample Log Table". For a more detailed explanation of the Enviroquant software, consult the appropriate manual, Organic Department Manager, or senior chemist within the GC/MS group.

The DFTPP solution must be analyzed once at the beginning of each twelve hour period during which standards and/or samples are analyzed. The 12 hour time period for GC/MS system begins at the moment of injection of the DFTPP analysis. The time period ends after twelve hours has elapsed according to the system clock. The last injection must be accomplished prior to the expiration of 12 hours; conceivably, the run-time of an injection could end after the twelve hours.

When the DFTPP has concluded, the run must be evaluated to determine if sample analysis can proceed. The chromatography and the ion ratios must be examined. The DFTPP run is processed using the current algorithms in the Target software.

The DFTPP tuning standard should also be used to assess the column performance and injection port inertness. Calculate the degradation of DDT to DDE and DDD; it should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, with no evidence of peak tailing. For clients requiring DOD criteria, the tailing factors for these two compounds should not exceed 2.

In order to document the performance of benzidine, pentachlorophenol and DDT, the following procedure must be followed. At the PC, which operates the instrument, load the method TUNETAIL.M into the ENVDA screen. Go into the quant drop down menu and select *calculate/generate report*. When that finishes, select *Qedit quant result.* Each compound can now be evaluated. Double click on benzidine and select *ChromEval* and then *Evaluate tailing*. Follow the instructions given on the screen to evaluate tailing. Send the report to the printer. Repeat the procedure for pentachlorophenol. Repeat the procedure for DDT, selecting *Evaluate degradation*. Follow the instructions given on the screen and then send the report to the printer. The report should be filed with the tune raw data.

If the results indicate the system does not meet acceptance criteria, the GC/MS must be manually tuned. Once the manual tune procedure is completed, DFTPP must be re-injected and reevaluated. If the instrument still does not meet criteria, notify your Department Manager. Under no circumstances should calibration proceed if the instrument DFTPP is not in criteria.

7.5 INSTRUMENT CALIBRATION

7.5.1 Initial Calibration for Method 8270-SIM

Prior to the analysis of samples and required method blanks, and after the instrument DFTPP tuning criteria have been met, the GC/MS system must be calibrated at six different concentrations, typically, 0.20, 0.50, 1.0, 2.0, 5.0 and 8.0 ng/uL. This is done to determine instrument sensitivity and the

linearity of GC/MS response for the semivolatile target and surrogate compounds.

Some SIM compounds may need to be calibrated at higher concentrations. A second standard is prepared containing these compounds. The two standards are combined as in the example below. The full aliquot is used and spiked with the appropriate amount of IS.

Example –

For a calibration at the following levels:
Calibration mix A would be prepared containing ALL analytes at 20 ng/ul
Calibration Mix B would be prepared containing phenols and phthalates at 20 ng/ul.

Final PAH conc. (ng/uL)	Final Conc. Phenols and phthalates (ng/ul)	Cal-Mix A Added (uL)	Cal-Mix B Added (uL)	$MeCl_2$ Added (uL)	Final Volume (uL)
0.20	1.0	10	40	950	1000
0.50	2.0	25	75	900	1000
1.0	3.0	50	100	850	1000
2.0	4.0	100	100	800	1000
5.0	5.0	250	0	750	1000
8.0	8.0	400	0	600	1000

Note: Calibration Mix B only is used to boost the phenols and phthalates concentrations in Cal. levels 1 through 4.

The GC/MS operating conditions for the calibration standards injections are the same as for the DFTPP with the following exceptions:

GC/MS Operating Conditions – Calibration and Samples	
Column temperature program	40°C for 3 min. to 300°C at 10°/min.
Final column temperature hold	300°C
Run time	35 minutes (time may vary dependent upon column length)
Scan start time	2.0-6.0 minutes (time may vary dependent upon column length)
Sample volume	2 uL

The conditions are set up in the method file LSPSIMXX.M
After analysis of the six calibration points, they must be quantitated and evaluated for adherence to QC criteria. Minimum requirements of ID files are the use of specific quantitation ions and quantitating a specific set of targets and surrogates with a set internal standard. Of particular importance when

performing SIM analysis are the ion ratios. These requirements are found in Tables 3 and 5.

7.5.2 Initial Calibration Criteria

Relative response factors (RRFs) must be calculated and evaluated for each target compound and surrogate. The RRF is defined as follows:

$$RRF = \frac{Ax}{A_{IS}} \times \frac{C_{IS}}{Cx}$$

where: Ax = area of the primary ion for the target compound
A_{IS} = area of the primary ion for the corresponding istd
C_{IS} = concentration of the istd (ng/uL)
C_x = concentration of the target compound

After the calibration points have been quantitated, update the calibration curve points using the Target data processing software to generate the RRF's and %RSD's for all analytes. If information is needed concerning the use of these programs, consult the Organic Department Manager or a senior chemist within the group.

Response factor criteria have been established for the calibration of the semivolatile target and surrogate compounds. These criteria must be met in order for the calibration curve to be considered valid. The percent RSD for each calibration check compound (CCC) must be less than or equal to 30 percent. There are three CCC's: Acenaphthene, Fluoranthene, and Benzo(a)pyrene. There are no criteria for the SPCC compounds. This is also applicable to clients that request DOD criteria.

7.5.2.1 Linearity of Target Analytes (This is also applicable to clients that request DOD criteria.)

If the RSD of any target analyte is 15% or less, then the response factor is presumed to be constant over the calibration range, and the average response factor may be used for quantitation.

If the RSD of any target analyte exceeds 15%, then a calibration option outlined in section 7.0 of method 8000 will need to be employed.

Option 1 (Section 7.5.2 of method 8000 - Rev. 2, 12/96), is a linear regression of instrument response versus the standard concentration. The correlation coefficient (r) for each target analyte and surrogate must be greater than or equal to 0.995. Target software calculates the correlation coefficient squared (r^2). This must be equal to or greater than 0.990.

Option 2 (Section 7.5.3 of method 8000 - Rev. 2, 12/96), is a non-linear calibration model not to exceed a third order polynomial. The lab would use a quadratic model or second order polynomial. The use of a quadratic model requires six calibration points. In order for the quadratic model to be acceptable, the coefficient of determination must be greater than or equal to 0.990.

If time remains in the clock after meeting the initial calibration acceptance criteria, samples may be analyzed. The calibration must be verified each twelve hour time period (time period starts from the moment of the DFTPP injection) for Method 8270-SIM. The SSTD1.0 in the curve may be used as the continuing calibration standard as long as it meets the continuing calibration acceptance criteria. All sample results must be quantitated using the initial calibration response factors.

7.5.2.2 Immediately following calibration an Independent Calibration Verification Standard must be analyzed. For clients requiring DOD criteria, all project analytes must be within +/- 20% of true value.

7.5.3 Continuing Calibration

A check of the calibration curve must be performed once every twelve hours immediately following analysis of the tuning compound DFTPP. This check contains all target compounds and surrogates at a concentration of 1.0 ng/uL.

After quantitation of the 1.0 ng/uL continuing calibration check, response factors must be calculated and compared to the average response factors in the initial calibration. The Target program calculates the calibration check response factors and compares them to the average RFs in the calibration curve by calculating percent differences. The method 8270 CCC's must have a % difference of +/- 20%D in order to be considered in criteria. These conditions must be met before method blank and/or sample analysis can begin. For clients requiring DOD criteria, all project analytes and surrogates must be within +/- 20%.

If the continuing calibration check does not meet criteria, corrective action must be taken. Depending on the situation, corrective action may be as follows:

- Re-analyze the 1.0 ng/uL continuing calibration check.
- Change the septum; clean the injection port; install a clean, silanized quartz liner; cut off a small portion (1" to 3") of the front end of the capillary column. This is usually performed when chromatography is poor. Record any of these actions in the appropriate instrument maintenance logbook.
- Analyze a new initial calibration curve.

The last option, the generation of a new initial calibration curve, is usually chosen when percent difference are >30%. In these instances, there is little or no chance of a continuing calibration reanalysis meeting criteria. If there is any doubt concerning which corrective action to undertake, consult the Organic Department Manager or a senior chemist within the group.

If the continuing calibration does meet the criteria specified above then analysis may precede using initial calibration response factors.

7.5.4. Retention Time Windows

Retention time windows for each analyte and surrogate are set at the midpoint standard of the calibration curve, following every ICAL. On days when an ICAL is not performed, the initial CCV may be used. For each sample, the RRT shall be compared with the mid-point of the ICAL or the most recently updated RRT. If the RRT has changed by more than ± 0.006 RRT units indicates a significant change in system performance and the laboratory must take appropriate corrective action.

For projects or clients requiring DoD QSM 4.1, IS EICP areas must be within -50% to + 100% of the ICAL midpoint standard. The retention time must be ± 30 seconds from the retention time of the ICAL midpoint standard.

7.6 SAMPLE ANALYSIS

Sample extracts may be analyzed only after the GC/MS system has met tuning criteria, initial calibration and continuing calibration requirements. Ensure that the same instrument conditions are being used for tuning, calibration and sample analysis by reviewing the GC parameters using the “Edit entire method” option under the Method menu in MSTOP. Note that you can not edit a method if the instrument is running.

Extracts are stored in the refrigerator in the organics extraction laboratory at 4°C ±2°C. Remove them from the refrigerator and place them in the GC/MS laboratory semivolatile hood when ready for analysis.

Prepare a 1.8 mL clear glass vial (crimp top) with a disposable insert (350 uL). Add 100 uL of sample extract and 1.0 uL of the 80 ng/uL IS stock to the vial and then cap. This gives a 0.8 ng/uL final concentration for the internal standard compounds. The samples are topped with Teflon lined crimp top caps.

7.7 FINAL DATA PACKAGE

7.7.1 Initial Data Review (IDR)

The initial data review is accomplished by the analyst who ran the samples and is a review of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed sample. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed:

- Surrogate Recoveries
- Internal Standard Area Stability

- Method Blank Acceptance
- Chromatography
- Target Compound Detection/Quantitation/Review for false positives
- Laboratory Control Sample Recoveries
- Matrix Spike/Matrix Spike Duplicate Recoveries

The analyst must evaluate all data using the QA Acceptance Criteria table found within this SOP (Table 1). This table gives acceptance criteria and corrective actions for criteria that are not met. In addition to evaluating QC elements, the chromatography and quantitation of target analytes must be reviewed. During this review, the analyst checks the integration of each individual peak. The hardcopy has false positives crossed out so they can be reviewed for appropriateness by the Organic Department Manager.

7.7.2 Chromatography

The chromatography should be examined for the presence or absence of any ghost peaks and can also be used as an indication of whether or not matrix interferences might be affecting surrogate recoveries and/or istd area recoveries. Whether or not the chromatography is acceptable is a judgment call on the part of the analyst and should be used in conjunction with other monitored QC (e.g. surrogate recoveries) to determine the necessity of reanalyzing.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an “m” qualifier will automatically be printed on the quantitation report summary.

This manual integration package must then be submitted to the Department Manager or his/her designee, who will review each manual integration.

For specific manual integration procedures, refer to Katahdin SOP QA-812, "Manual Integration", current revision.

7.7.3 Target Compound Detection/Quantitation

The semivolatile ID files have been set up to err on the side of false positives; that is to identify and quantitate peaks as target compounds that may not necessarily be valid hits. It is the responsibility of the GC/MS analyst to use his/her technical judgment to determine if the identification of a target compound is correct or not.

If any target concentration exceeds the upper limit, a dilution must be made and analyzed. The dilution chosen should keep the concentration of the largest target compound hit in the upper half of the initial calibration range. LCS and MS/MSD samples need not be diluted to get spiked analytes within the calibration range.

The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity > 10% must be present in the sample spectrum.
- The relative intensities of primary and secondary ions must agree within ±20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum but not present in the standard spectrum must be considered and accounted for by the analyst.

If a compound cannot be verified by all three criteria above, but, in the technical judgment of the mass spectral interpretation specialist, the identification is correct, then the laboratory shall report that compound on the Form 1 as a valid hit.

The GC/MS laboratory initial data review must be completed within twelve hours of batch completion; in the majority of instances, the initial review should be accomplished at the beginning of a work shift for the previous set of analyses.

7.7.4 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into QuickForms. Depending on the QC label requested by the client, a Report

of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the department manager for final review. A complete review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

7.8 Injection Port Liner Cleaning And Silanizing Procedure

- Remove the rubber o-ring from the liner and place the liner in a large Erlenmeyer flask.
- In the hood, pour nitric acid into the flask until the liner is covered. Place the flask on a hotplate and boil for 2-3 hours.
- Let cool; drain nitric acid and thoroughly flush the liner with water.
- Bake briefly in the muffle oven until liner is dry and cool to room temperature.
- Place the liner in a beaker, fill with Sylon and let it soak for at least two hours.
- Take out the liner and rinse it thoroughly with toluene.
- Rinse the liner thoroughly with purge and trap grade methanol.
- Bake the liner in the muffle oven for a minimum of three hours.

7.9 Instrument Maintenance

Instrument preventative maintenance is performed on a semi-annual basis by GC/MS chemists. This maintenance includes a thorough inspection and cleaning of all parts, including changing rough and turbopump oils. GC/MS analysts perform other maintenance on an as-needed basis. Typically, routine maintenance involves clipping off the front end of the DB-5MS column, replacing the injection port septum, and installing a freshly silanized quartz liner after sample analysis.

All maintenance must be documented in the instrument-specific maintenance log, whether it is routine or not. The Department Manager must authorize any maintenance over and above a routine source cleaning.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are

based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 Method Blank Criteria

A method blank is defined as a volume of a clean reference material (deionized distilled water for water samples, baked organic-free sand for soil/sediment matrices) that is carried through the entire analytical procedure. One method blank must be extracted with each group of samples of a similar matrix and must be analyzed on the GC/MS system that was used to analyze the samples.
An acceptable method blank must contain less than or equal to the PQL of any target compound. For clients requiring DOD criteria, no analytes detected at > ½ PQL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit.

If the method blank exceeds these contamination levels, the analytical system is considered out of control and corrective action must be taken before sample analysis.

Reanalysis of the blank is the first step of the corrective action; if that does not solve the problem, a Katahdin Corrective Action Report (CAR) will be initiated. Corrective action will be specified after consultation including the Department Manager, Operations Manager, and QA Officer.

8.2 Surrogate Recoveries

The five surrogates (2-Methylnaphthalene-d10, 2,4-Dibromophenol, Fluorene-d10, Pyrene-d10 and 1,4-Dioxaned8) must meet the current statistically derived or nominal acceptance limits. If statistical limits have not been established then the surrogate recovery must meet the nominal limits of 30-150%. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

If specifications are not met, the sample (or blank) should be reanalyzed. If specifications are met in the reanalysis, this reanalysis should only be submitted. If

surrogate specifications are not met in the sample or method blank reanalysis, a Corrective Action Report (CAR) should be initiated. Corrective action will be specified after consultation including the Department Manager and Operations Manager.

For further information regarding the acceptance of surrogate recoveries, consult the Organic Department Manager.

8.3 Internal Standard Responses

Internal standard responses and retention times (RT) in all samples and blanks must be evaluated as part of the technical data review. The method files have been set up to only detect compounds that fall within a set RT window. For Method 8270-SIM analysis, if the extracted ion current profile (EICP) area for any internal standard changes by more than a factor of two (-50% to +100%) as compared to the daily continuing calibration standard, reanalysis must occur. If the reanalysis meets criteria, only the in-criteria run should be reported. If the reanalysis is still out of criteria, both analyses should be included in the sample package set.

For projects or clients requiring DoD QSM 4.1, IS EICP areas must be within -50% to + 100% of the ICAL midpoint standard. The retention time must be ± 30 seconds from the retention time of the ICAL midpoint standard.

MS/MSD samples that do not meet the EICP area criteria above do not have to be reanalyzed.

8.4 Laboratory Control Sample (LCS)

An LCS must be performed for each group of samples of a similar matrix, for the following, whichever is more frequent:

- Every 20 samples of a similar matrix or similar concentration, or
- Every batch of samples extracted.

Statistical limits are compiled annually for LCS recoveries (archived in QA office). Statistical limits are only calculated when at least 20 usable data points are obtained for any given compound. If insufficient data points are available, nominal limits are set by the section supervisor, Laboratory Operations Manager and Quality Assurance Officer. Refer to Katahdin SOP QA-808, “Generation and Implementation of Statistical QC Limits and/or Control Charts”, current revision.

The use of statistical limits versus nominal limits is dependent on the client and project. This information is communicated to the Organic Department Manager through the Katahdin project manager. It is standard practice to use statistical limits for reporting purposes and to evaluate any QC criteria exceedances. However, nominal limits of 30-150% may be used for some projects or states (i.e. South Carolina). For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

The LCS recoveries for all analytes are evaluated. All of the compounds of interest must fall within the established statistical limits or nominal limits with the following sporadic exceedance allowances, for DoD clients.

# of Analytes	# of Allowable Exceedances
> 90	5
71 – 90	4
51 – 70	3
31 – 50	2
11 – 30	1
<11	0

If less than the number of allowable exceedances fail the statistical limits, no corrective action is needed. If greater than the number of allowable exceedances fail the statistical limits, corrective action may be taken. Corrective actions may vary with each situation. However, in the case where the failures are high and the samples are non-detect for those compounds, then no corrective action is required. Otherwise, corrective action may involve reanalysis or recalibration. The specific corrective actions taken will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time.

For non-DoD clients corrective action is only taken if greater than 10% of the analytes of interest are outside of the laboratory established acceptance limits.

8.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Criteria

Matrix Spike and Matrix Spike Duplicates must be extracted and analyzed for each group of up to 20 samples of a similar matrix or similar concentration. In the event insufficient sample volume is available an LCS/LCS Duplicate is extracted and analyzed in place of the MS/MSD.
Statistical limits are compiled annually for MS/MSD recoveries for a short list of the spiked compounds (Acenaphthene, Pentachlorophenol and Pyrene). Nominal limits of 30-130% are used for all other compounds. Generally, corrective action is only taken for the short list of the spiked compounds. The specific corrective actions will rely on analyst experience to make sound scientific judgments while considering client objectives, other quality control indicators and/or the ability to reanalyze a sample within holding time. For clients requiring DOD criteria, use acceptance limits specified by DOD or use in-house limits where none are specified.

A Corrective Action Report (CAR) must be filled out and filed if any criteria for percent recovery or relative percent difference are not met to document any decisions with reporting data.

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9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 8270 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846, 3rd edition, Final Updates I, II, IIA, IIB, III, IIIA, and IIIB, Nov 2004, Method 8270C.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 8270D.

Department of Defense Quality Systems Manual for Environmental Laboratories (DoD QSM), Current Version.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

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The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

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TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Check of mass spectral ion intensities using DFTPP	Prior to initial calibration and calibration verification	Refer to the criteria listed in Section 7.4	Retune instrument, and verify
Six-point initial calibration for all analytes	Initial calibration prior to sample analysis	RSD ≤30 for RFs of the CCCs; Average %RSD < 15% for all compounds. Refer to section 7.5.2.1 for more details.	Repeat calibration if criterion is not met
Independent calibration verification	Once after Initial calibration	± 20 % D	1) Reanalyze standard 2) Reprep standard 3) Reprep standard from fresh stock.
Continuing calibration verification	Once per each 12 hours, prior to sample analysis	CCCs ≤ 20%D	Repeat initial calibration and reanalyze all samples analyzed since the last successful calibration verification
ISs	Immediately after or during data acquisition of calibration check standard	Retention time ± 30 seconds; EICP area within -50% to +100% of last calibration verification (12 hours) for each IS	Inspect mass spectrometer or GC for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning
Demonstration of ability to generate acceptable accuracy and precision	Once per analyst initially and annually thereafter	All recoveries within method QC acceptance limits.	Recalculate results; locate and fix problem; reextract/reanalyze P&A study for those analytes that did not meet criteria
Method blank	One per prep batch	No analytes detected > PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e.If the blank results are above the PQL, report samples that are <PQL or > 10X the blank result. Reprep a blank and the remaining samples.
LCS for all analytes	One LCS per prep batch	Statistically derived from lab data or nominal limits depending on the project. . See also section 8.4 of this SOP for more information on allowable exceedances.	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. If an LCS/LCSD was performed and only one was unacceptable, narrate. If the surrogate recoveries in the LCS are low but are acceptable in the blank and samples, narrate. If the LCS rec. is high but the sample results are <PQL, narrate. Otherwise, reprep a blank and the remaining samples.

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TABLE 1 (cont.)

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogate spike	Every sample, control, standard, and method blank	Statistically derived limits.	(1) Check chromatogram for interference; if found, flag data (2) If not found, check instrument performance; if problem is found, correct and reanalyze (3) If still out reextract and analyze sample (4) If reanalysis is out, flag data
MS/MSD	One MS/MSD per every 20 samples	Statistically derived from lab data or nominal limits depending on the project. Nominal limits are used as default limits.	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep the samples and QC.
MDL and/or LOD/LOQ Verification study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

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TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to method for specific ion criteria.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	Correct problem then repeat breakdown check.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2.	At the beginning of each 12-hour period, prior to analysis of samples.	Flagging criteria are not appropriate.	No samples shall be run until degradation ≤ 20%.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs ≥ 0.050. 2. RSD for RFs for CCCs ≤ 30% and one option below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression r ≥ 0.995; Option 3: non-linear regression– coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value.	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 2 (cont.)

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	Laboratories may update the retention times based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping). With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ±0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs ≥ 0.050. 2. %Difference/Drift for all target compounds and surrogates ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration).	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since last acceptable CCV.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL; EICP area within -50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply Q-flag to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 2 (cont)

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS CLs, if available depending on project requirements. In-house CLs may not be greater than ± 3 times the standard deviation of the mean LCS recovery. A number of analytes may fall outside the CL but within marginal exceedance limit depending on the total number of analytes in the LCS.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Refer to Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix if sufficient sample is available.	The laboratory shall use laboratory LCS CLs or use DoD-generated LCS CLs, if available depending on project requirements.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix if sufficient sample is available.	MSD: For matrix evaluation, use laboratory LCS CLs or use DoD-generated LCS CLs, if available depending on project requirements. MS/MSD: RPD ≤ 30%.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 2 (cont)

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	The laboratory shall use laboratory surrogate CLs or use DoD-generated surrogate CLs, if available depending on project requirements. .	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepped within hold time.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-213-11	METHOD 8270, current revision
Apparatus/Materials	none	
Reagents	none	
Sample preservation/ handling	none	
Procedures	none	
QC - Spikes	none	
QC - LCS	none	
QC - Accuracy/Precision	none	
QC - MDL	none	

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 4

ANALYTE QUANITIATION AND INTERNAL STANDARDS

Internal Standard: 1,4-dichlorobenzene-d4	2,4-Dinitrotoluene
Target and Surrogates:	2,4-Dinitrophenol
1,4-Dioxane	2,3,4,6-Tetrachlorophenol
1,4-Dioxane-d8 (surrogate)	Diethylphthalate
Benzaldehyde	4-Chlorophenyl-phenyl ether
Phenol	4,6-Dinitro-2-methylphenol
bis(2-Chloroethyl)ether	N-nitrosodiphenylamine
2-Chlorophenol	2-Nitroaniline
2-Methylphenol	3-Nitroaniline
3&4-Methylphenol	4-Nitroaniline
2,2'-Oxybis(1-chloropropane)	Dibenzofuran
Nitrobenzene	4-Nitrophenol
Hexachloroethane	**Internal Standard: Phenanthrene-d10**
Acetophenone	Target and Surrogates:
N-nitroso-di-n-propylamine	Pentachlorophenol
Internal Standard: Naphthalene-d8	1-Methylphenanthrene (dredge)
Target and Surrogates:	Phenanthrene
Naphthalene	Hexachlorobenzene (special)
1-Methylnaphthalene (dredge)	Anthracene
2-Methylnaphthalene	Fluoranthene
2-Methylnaphthalene-D10 (surrogate)	Carbazole
Isophorone	Di-n-butylphthalate
2-Nitrophenol	4-Bromophenyl-phenyl ether
2,4-Dimethylphenol	Atrazine
bis(2-Chloroethoxy)methane	**Internal Standard: Chrysene-d12**
2,4-Dichlorophenol	Target and Surrogates:
4-Chloroaniline	Butylbenzylphthalate
Hexachlorobutadiene	3,3'-Dichlorobenzidine
Caprolactam	Pyrene
4-Chloro-3-methylphenol	Benzo(a)Anthracene
Internal Standard: Acenaphthene-d10	Chrysene
Target and Surrogates:	Bis-(2-ethylhexyl)phthalate
1,1'-Biphenyl (dredge)	Pyrene-d10 (surrogate)
2,6 Dimethylnapthalene (dredge)	**Internal Standard: Perylene-d12**
Acenaphthylene	Target and Surrogates:
Acenaphthene	Perylene (dredge)
Fluorene	Benzo(b)fluoranthene
2-Fluorene-d10 (surrogate)	Benzo(k)fluoranthene
2,4-Dibromophenol (surrogate)	Benzo(e)pyrene (dredge)
2-Chloronaphthalene	Di-n-octylphthalate
Hexachlorocyclopentadiene	Benzo(a)pyrene
2,4,6-Trichlorophenol	Indeno(1,2,3-cd)pyrene
2,4,5-Trichlorophenol	Dibenz(a,h)anthracene
Dimethylphthalate	Benzo(ghi)perylene
2,6-Dinitrotoluene	

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 5

PROCEDURE CONDENSATION

Clock

12 hours from injection of 50ng DFTPP.

Calibration Curve Criteria

<30% RSD for CCCS
<15% RSD average for all analytes in calibration standard

Continuing Calibration Check Criteria

<20% D for CCC compounds

Additional QC

LCS every extraction batch
MS/MSD every 20 samples

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 6

SVOA COMPOUNDS AND CHARACERISTIC IONS

COMPOUND	PRIMARY ION	SECONDARY IONS
1,4-Dioxaned8	96	66
1,4-Dioxane	88	58
Benzaldehyde	77	105,106
Phenol	94	65,66
bis(2-Chloroethyl)ether	93	63,95
2-Chlorophenol	128	64,130
1,4-Dichlorobenzene-d4 (IS)	152	150,115
2,2'-Oxybis(1-choropropane)	45	77,121
2-Methylphenol	108	107,77
Acetophenone	105	77,51
N-nitroso-di-n-propylamine	70	52,101
Hexachloroethane	117	201,199
3&4-Methylphenol	108	107,77
Nitrobenzene	77	123,51
Isophorone	82	54,138
2-Nitrophenol	139	109,81
2,4-Dimethylphenol	107	122,121
bis(2-Chloroethoxy)methane	93	63,123
2,4-Dichlorophenol	162	164,98
Naphthalene-d8 (IS)	136	137,134
Naphthalene	128	129,127
4-Chloroaniline	127	129
Hexachlorobutadiene	225	223,227
Caprolactam	113	55,56
4-Chloro-3-methylphenol	107	77,142
2,4-Dibromophenol (surr)	252	63,143
2-Methylnaphthalene-d10 (surr)	152	150
2-Methylnaphthalene	142	141,115
1-Methylnaphthalene	142	141,115
Hexachlorocyclopentadiene	237	235,239
2,4,6-Trichlorophenol	196	198,200
2,4,5-Trichlorophenol	196	198,200
2-Chloronaphthalene	162	127,164
1,1'-Biphenyl	154	153,76
2-Nitroaniline	65	92,138
Dimethylphthalate	163	194,164
2,6-Dinitrotoluene	165	63,89
Acenaphthylene	152	151,153
Acenaphthene	152	154,152
Acenaphthene-d10 (IS)	164	162
3-Nitroaniline	138	65,92
2,4-Dinitrophenol	184	107
Dibenzofuran	168	139

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

TABLE 6 (cont.)

SVOA COMPOUNDS AND CHARACERISTIC IONS

2,4-Dinitrotoluene	165	63
4-Nitrophenol	109	139,65
2,3,4,6-Ttrachlorophenol	232	230
Diethylphthalate	149	177,176
Fluorene-d10 (surr)	176	174,178
Fluorene	166	165
4-Chlorophenyl-phenyl ether	204	206,141
4-Nitroaniline	138	108,65
4,6-Dinitro-2-methylphenol	198	121
N-nitrosodiphenylamine	169	168,167
4-Bromophenyl-phenyl ether	248	250,141
Hexachlorobenzene	284	142,249
Atrazine	200	173,215
Pentachlorophenol	266	264,268
Phenanthrene-d10 (IS)	188	189
Phenanthrene	178	179,176
Anthracene	178	179,176
Carbazole	167	166,139
Di-n-butylphthalate	149	150,104
Fluoranthene	202	200,203
Pyrene	202	200,201
Pyrene-d10 (surr)	212	210,106
Butylbenzylphthalate	149	91,206
Benzo(a)anthracene	228	229,226
Chrysene-d12 (IS)	240	236,120
3,3-Dichlorobenzidine	252	254,126
Chrysene	228	226,229
bis(2-Ethylhexyl)phthalate	149	167
Di-n-octylphthalate	149	150
Benzo(b)fluoranthene	252	253,125
Benzo(k)fluoranthene	252	253,125
Benzo(a)pyrene	252	253,250
Perylene-d12 (IS)	264	260
Indeno(1,2,3-cd)pyrene	276	277
Dibenzo(a,h)anthracene	278	279
Benzo(g,h,i)perylene	276	277

Primary ions must not be changed except in unusual instances where interference occurs with a co-eluting non-target analyte. In this case, a secondary ion may be used for quantitation with the following rules:

(1) The corresponding standard(s) (initial calibration curve and continuing calibration standard) must be re-quantitated with the secondary ion.
(2) Approval must be obtained from the Organic Department Manager or the laboratory Operations Manager.

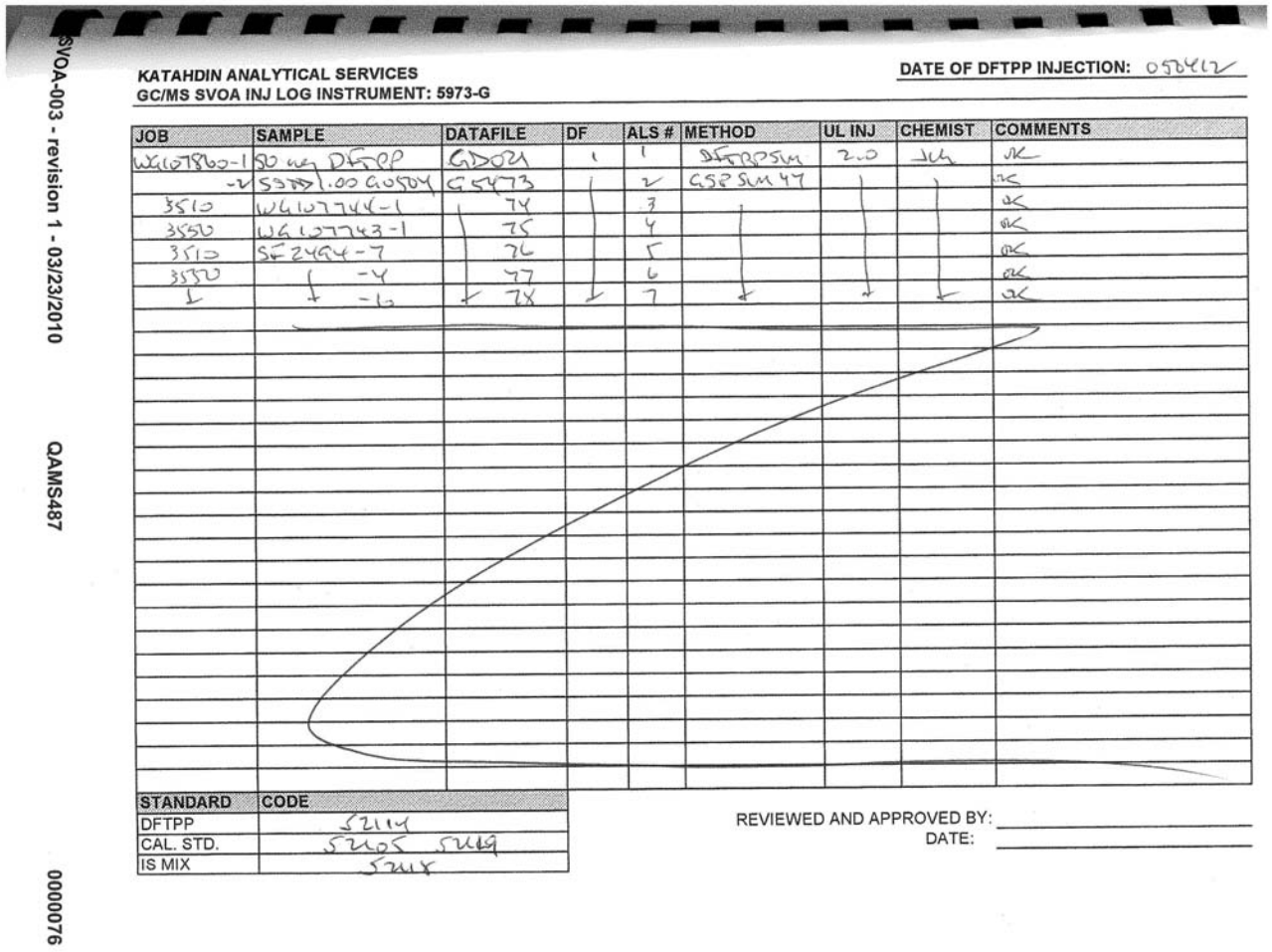
The quantitation ion must then be changed back to the one specified in the table above after quantitation of the samples(s).

Secondary ions are recommended only and may be changed depending upon instrument conditions (sensitivity, etc.). However, it is Katahdin policy that a minimum of 2 ions (primary and one secondary) be used for all GC/MS analyses.

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 1

EXAMPLE OF RUNLOG LOGBOOK PAGE



SVOA-003 - revision 1 - 03/23/2010

QAMS487

0000076

KATAHDIN ANALYTICAL SERVICES
GC/MS SVOA INJ LOG INSTRUMENT: 5973-G

DATE OF DFTPP INJECTION: 05/04/12

JOB	SAMPLE	DATAFILE	DF	ALS #	METHOD	UL INJ	CHEMIST	COMMENTS
WG107863-1	50 ng DFTPP	GD021	1	1	DFTPPSIM	2.0	JK	JK
-2	5 ng 1.00 GC0504	G5473		2	GSP SIM 47			JK
3510	WG107744-1	74		3				JK
3550	WG107743-1	75		4				JK
3510	SF 2494-7	76		5				JK
3550	-4	77		6				JK
	-6	78		7				JK

STANDARD	CODE
DFTPP	52114
CAL. STD.	52105 52109
IS MIX	52117

REVIEWED AND APPROVED BY: _____
DATE: _____

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

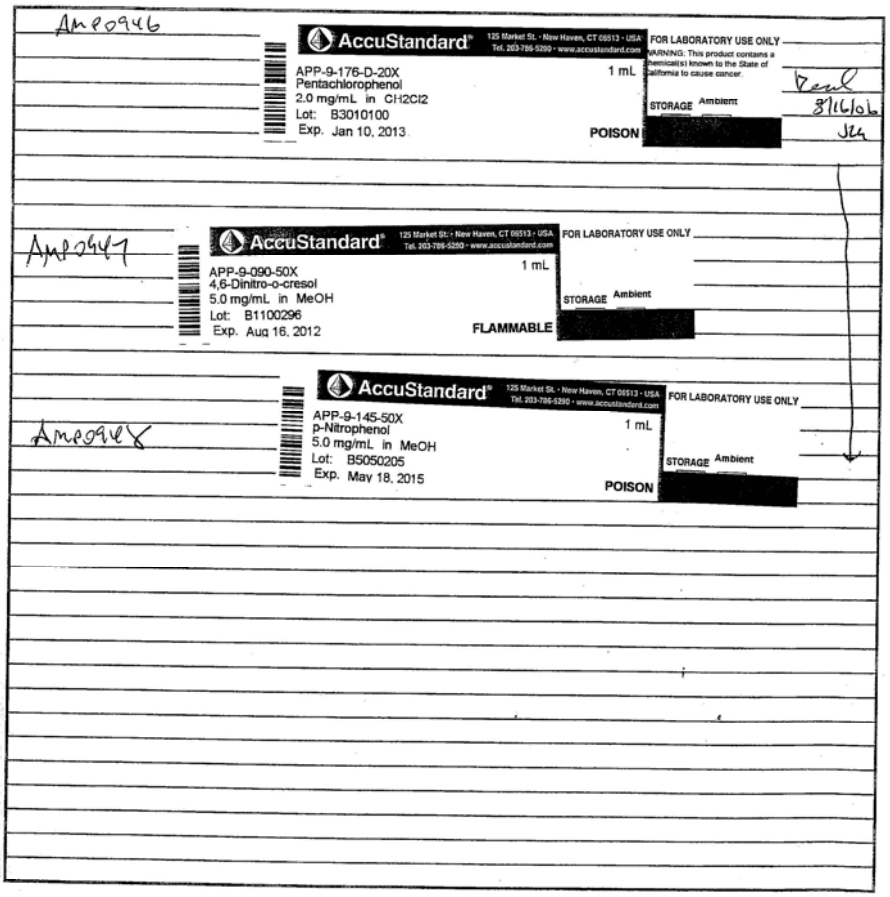
FIGURE 2

EXAMPLE OF GC/MS STANDARDS RECEIPT LOGBOOK ENTRY

KATAHDIN ANALYTICAL SERVICES

STOCK STANDARDS RECEIVED

GCMS LABORATORY
REVIEWED BY/DATE:



QAMS294

0000045

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

FIGURE 3

EXAMPLE OF SVOA STANDARDS PREPARATION LOGBOOK ENTRY

GC/MS SVOA STANDARD PREP LOGBOOK

50863	8270 Stock (w/o MeOH)	3-15-06	7-7-06	JLM	AMP0884	8270 Megamix	300	2-22-07	4.2ml	150 ug/ml
					AMP0937	"	350	3-15-07		
					AMP0911	APP IX #2	600	3-2-07		
					AMP0910	" 1	180	3-9-07		
					AMP0570	" 1	200	7-7-06		
					AMP0699	Organophosphate	300	8-19-06		
					AMP0931	Benzoic Acid		3-9-07		
					AMP0907	Hexachlorophene		2-22-07		
					AMP0906	Benzidine		3-9-07		
					AMP0936	3,3'-Dichlorobenzidine		3-14-07		
					AMP0932	8270 Surr	150	3-9-07		
					50861	DCA	300	3-13-07		
					B43590	MeCl2	550	~		
50864	8270 level 1	3-15-06	7-7-06	JLM	50863	8270 Stock	70	7-7-06	1.05ml	10 ug/ml
					B43590	MeCl2	980			
50865	8270 level 2	3-15-06	7-7-06	JLM	50863	8270 Stock	150	7-7-06	0.90ml	25 ug/ml
					B43590	MeCl2	750			
50866	8270 level 3	3-15-06	7-7-06	JLM	50863	8270 Stock	600	7-7-06	1.8ml	50 ug/ml
					B43590	MeCl2	1200			
50867	8270 level 4	3-15-06	7-7-06	JLM	50864	8270 Stock	700	7-7-06	1.05ml	100 ug/ml
					B43590	MeCl2	350			

Reviewed by/Date:

CAMS2284

0000057

TITLE: ANALYSIS OF SEMIVOLATILE ORGANIC COMPOUNDS BY: SW 846 METHOD 8270 – Modified for Selected Ion Monitoring (SIM)

ADDENDUM 1

LOW LEVEL 1,4-DIOXANE ANALYSIS

The following are differences from the standard 8270 C or D SIM analysis:

GC Operating Conditions -

GC/MS Operating Conditions – Calibration and Samples	
Column temperature program	35°C for 4 min. to 300°C for 12°/min.
Final column temperature hold	300°C
Run time	32 minutes (time may vary dependent upon column length)
Scan start time	2.0-3.0 minutes (time may vary dependent upon column length)
Sample volume	2 uL

Stock Standards – 1,4-Dioxane and 1,4-Dioxane each at 20 ug/mL

Calibration Standards – Use the above stock standards to prepare calibration standards at concentrations 0.25 ug/mL, 0.50 ug/mL, 1.0 ug/mL, 2.0 ug/mL, 4.0 ug/mL and 6.0 ug/mL. The 1.0 ug/mL is also the continuing calibration verification standard.

Sample analysis – Add 1 uL of internal standard (Section 5.3.2.4) aliquot of sample.

The ions for 1,4-Dioxane are 96 and 64.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Prepared By: Mike Thomas Date: 8/96

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: JCBurton Date: 10/25/00

QA Officer: Deborah J. Nadeau Date: 10.23.00

General Manager: Dennis J. Hulgan Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Miner changes throughout. Clarifications to procedure section.	DN	10.23.00	
02	Addition of SPE Propedure. Minor changes throughout Added wording to Sections 6 and 8	LAD	013105	013105
03	Added separate QC for Pest. and PCB. updated concentration procedure to reflect current practices. Changes in wording for clarification. Update Logbook page	LAD	04/06	04/06
04	Added waste generated and disposal info. Added missing definitions. Updated SPE extraction procedure. Updated Table 1 and 2. Added Table 3.	LAD	09/07	09/07
05	Updated logbook example. Added logbook requirements	LAD	09/08	09/08

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added information for determining initial volume. Added reference to CA-108. Added clarification for LCS/D and MS/D sets for PEST/PCB analysis. Minor changes to reflect current techniques.	LAD	10/09	10/09
07	Added additional solvent exchange procedure. Updated logbook example.	LAD	08/10	08/10
08	Added that a MS/MSD should be extracted if enough sample volume and to extract an LCSD if no MS/D. Added wording to H_2SO_4 prep. Minor changes to reflect current practices and remove duplication. Updated MDL-Sect. 9. Added and updated references. Removed method 3535 throughout. Changed PCB H.T to 30 Days w/ explanation.	LAD	04/12	04/12
09	Sect. 7- Removed the procedure of marking the sample meniscus on the sample bottle with a grease pencil.	LAD	05/13	05/13

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy _____ of document **SOP CA-515-09,** titled **PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy _____ of document **SOP CA-515-09,** titled **PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS**.

Recipient: _____ Date: _____

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedures utilized by Katahdin Analytical Services, Inc. laboratory personnel for the preparation of aqueous samples prior to analysis for pesticides/PCBs by GC/ECD. It includes extraction of water samples by separatory funnel and continuous liquid-liquid extraction methods (EPA Methods 3510 and 3520.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in the extraction of aqueous samples for pesticides/PCBs analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training”.

It is the responsibility of all Katahdin personnel involved in the preparation of aqueous samples for pesticides/PCBs analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the

samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for the data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Management Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. This includes the methylene chloride waste layer generated during CLLE extraction. Special care should be taken to pour this layer off into the appropriate waste stream, leaving the sample waste to be disposed of as follows. Since Pesticide/PCB samples are at a neutral pH, SEP funnel or CLLE sample waste may be dumped into either the “N-Hi” or “N-low” satellite accumulation area. Acetone and hexane are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the “O” satellite accumulation area nearest the point of generation. Please refer to

the current revision of SOP CA-107 for the location of satellite waste accumulation areas

2.0 SUMMARY OF METHOD

Pesticides/PCBs are extracted from aqueous samples using methylene chloride and separatory funnel or continuous liquid-liquid apparatus, following EPA Methods 3510 and 3520. The methylene chloride is exchanged with hexane for the final extract. Method detection limit studies must be performed annually for pesticides/PCBs using all extraction methods, if the extraction lab wishes to use either or all techniques. Method 3510 (separatory funnel) is generally preferred for pesticides/PCBs since organochlorine pesticides may dechlorinate if under elevated pH conditions for an extended period of time. (Section 3.2, Method 3510B, Rev. 2, 9/94)

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates which are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves which have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or Teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

4.0 APPARATUS AND MATERIALS

Prior to use, all glassware must be rinsed three times with the solvent to be used for extraction.

4.1 Separatory Funnel - 2000 mL capacity, Nalgene Teflon FEP separatory funnels with Nalgene Tefzel® screw-cap closures (or equivalent)

4.2 Concentrator tube - 10 mL, graduated

4.3 Evaporative flask - Kuderna-Danish, 500 mL capacity attached to concentrator with neck clips

4.4 Snyder column - Kuderna-Danish, three ball macro

4.5 Graduated cylinders - 100 mL, 1000 mL, or 2000 mL

4.6 Short Stem Funnels

4.7 250 mL amber collection bottles with Teflon-lined caps

4.8 12 mL and/or 16 mL glass vials with Teflon-lined caps

4.9 Continuous liquid-liquid extractors (CLLE) including body, 500 mL flat bottom boiling flask and Alhin condensers

4.10 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.11 Nitrogen evaporation apparatus.

4.12 Boiling chips - approximately 10/40 mesh, Teflon or selenized carborundum, 12 mesh (or equivalent). Cleaned by Soxhlet.

4.13 Water bath - eight position concentric ring bath or equivalent, equipped with a calibrated thermometer.

5.0 REAGENTS

5.1 Laboratory reagent grade water - water in which an interferent is not observed at or above the PQL for any parameter of interest (carbon filtered ASTM Type II water or equivalent)

5.2 Sodium Hydroxide (10N) – Purchased from vendor, “Baker-analyzed”, or equivalent

5.3 Sodium Sulfate (ACS) - Granular, anhydrous. Bake at 400°C for 4 hours (may be done by vendor). Purify by rinsing three times with pesticide grade methylene chloride. Allow residual methylene chloride to evaporate before use. Stored in a Teflon capped glass bottle.

5.4 Sulfuric acid solution (1:1 H_2SO_4 : H_2O) – Prepared in an icebath by slowly adding a volume of concentrated H_2SO_4 to an equivalent volume of reagent water and swirl gently to mix. Caution should be taken when adding the acid to the water as the reaction is highly exothermic. Prepare as needed and store in a ground glass stoppered bottle.

5.5 Methylene Chloride ($MeCL_2$) - Pesticide grade or better. Lot must be verified by concentrating 300-400 mL to 1.0 mL and evaluating by GC/MS.

5.6 Acetone and Hexane - Pesticide grade or better. Lot must be verified by concentrating approximately 20-30 mL to 1.0 mL and evaluating by GC/ECD.

5.7 Pesticide/PCB Surrogate spiking solution - Prepare a solution of decachlorbiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1.0 ug/mL ea in acetone. Store the solution at –10 to -20 °C in a Teflon sealed container. Solution must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

5.8 Pesticide Matrix Spike/Lab Control Sample spiking solution - Prepare a matrix spiking solution in pesticide grade methanol that contains all target analytes listed below:

ANALYTE	ug/mL
4,4'-DDT	0.5
4,4'-DDD	0.5
4,4'-DDE	0.5
Aldrin	0.5
Dieldrin	0.5
Endrin	0.5
Endrin Aldehyde	0.5
Endrin Ketone	0.5
Endosulfan I	0.5
Endosulfan II	0.5
Endosulfan Sulfate	0.5
alpha-BHC	0.5
beta-BHC	0.5
delta-BHC	0.5
gamma-BHC (Lindane)	0.5
Heptachlor	0.5
Heptachlor epoxide	0.5
Methoxychlor	0.5
alpha-Chlordane	0.5
gamma-Chlordane	0.5

5.9 PCB Matrix Spike/Lab Control Sample spiking solution - Prepare a matrix spiking solution in pesticide grade acetone that contains 5.0ug/ml ea of Aroclor® 1016/1260 mix (Restek catalog# 32039).

5.10 Store the spiking solutions at –10 to -20 °C in a Teflon sealed container. The solutions must be verified by GC/ECD prior to use, and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples are collected in 1 L amber bottles and held at 4 (±2) °C until time of extraction.

The holding time for the extraction of aqueous pesticide samples for Methods 3510 and 3520 is 7 days from date of sample collection.

The holding time for the extraction of aqueous PCB samples by methods 3510 and 3520 is 30 Days

Note: SW-846 8082A does not specify a holding time to extraction; 30 days is presented here as a conservative limit. The method recommends a holding time of 40 days from extraction to analysis for extracts stored under refrigeration in the dark; but also refers to SW846 Chapter 4, which specifies that there is no holding time for PCBs. Additionally, SW-846 states that the holding times listed in the method under the conditions listed (apparently referring to storage of extracts) may be as long as a year.

Holding times may be dictated by a project specific Quality Assurance Project Plan (QAPP), in a program specific Quality Systems Manual (QSM) or by a regulating body. If a project requires a holding time that is not specified above it must noted in the analysis notes of the workorder.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- Sample pH if applicable
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Sample ID or QC sample ID
- Initial and final volumes or weight

- Surrogate and spike amounts
- Any sample cleanup preformed
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)
- Prep batch start time and end time
- CLLE start time and end time

SEPARATORY FUNNEL SAMPLE EXTRACTION

If an emulsion prevents acceptable recovery or client history indicates samples may demonstrate matrix interence, then samples should be extracted by continuous liquid-liquid extraction (CLLE).

7.1 Prerinse all glassware three times with methylene chloride prior to use.

7.2 Label a 2 L Teflon separatory funnel and a 250 mL amber collection bottle clearly. Label should include laboratory sample number, matrix, analyte, and extraction date. Be sure that the detachable stopcocks are secured to the separatory funnels before adding samples.

7.3 Measure the initial volume by comparing the meniscus of the sample with the reference bottle of the same bottle type. Please refer to SOP CA-108, “Basic Laboratory Technique”, for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook. Transfer the contents of the sample bottle to a 2 L separatory funnel.

7.4 Transfer 1 L of laboratory reagent grade water to a 2 L separatory funnel. This serves as a method blank for the extraction batch. A method blank must be prepared for every daily extraction batch of twenty or fewer samples.

7.5 Transfer 1 L of laboratory reagent grade water to a 2 L separatory funnel for each analysis to be performed (pesticide and/or PCB). This will serve as a Laboratory Control Sample (LCS). When Pesticides and PCBs are extracted together, a LCS and LCSD set must be extracted for each analysis. An LCS is required for every daily extraction batch of twenty or fewer samples and each analysis. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) is to be prepared as requested by a client or, at a minimum, one pair per 20 samples. An MS/MSD will be analyzed only if enough sample has been provided by the client. Additionally, in the event that the batch MS/MSD requirement cannot be fulfilled, a laboratory control spike duplicate must be analyzed. Transfer two additional 1 L aliquots of sample to 2 L separatory

funnels for a matrix spike and matrix spike duplicate (MS/MSD) for each analysis. When Pesticides and PCBs are extracted together, a MS and MSD set must be extracted for each analysis. Note: Sufficient sample volume should be available without depleting all remaining sample aliquots.

7.7 Check the pH of the samples. If it is not between pH 5 and 9, adjust the pH with 10N sodium hydroxide or 1:1 sulfuric acid solution. Note the addition of NaOH or H_2SO_4 in the extraction logbook.

7.8 Using a gas-tight syringe, add 1.0 mL of surrogate spiking solution to all samples the blank, LCS/LCSD(s) and MS/MSD(s), if performed.

7.9 Using a gas-tight syringe, add 1.0 mL of pesticide or PCB matrix spiking solution to the appropriate LCS, LCSD, MS and MSD if performed.

7.10 To each empty sample bottle add 60 mLs of methylene chloride, rinse the bottle and transfer the solvent into the appropriate separatory funnel. Add 60 mL of methylene chloride directly to the blank and LCS/LCSD(s).

7.11 Ensure that each screw cap is secured tightly to the separatory funnel to prevent leaks. Shake briefly and vent in hood to release pressure. Extract the sample by shaking the funnel on mechanical shaker for 3 minutes. Allow phases to separate for at least 10 minutes. Drain the methylene chloride layer into the 250 mL amber collection bottle.

7.12 If an emulsion forms, mechanical techniques must be employed to achieve maximum separation and solvent recovery. Such means include swirling and centrifugation and draining through a small separatory funnel. In certain instances, transferring the entire sample into a continuous liquid-liquid extractor may be the only alternative. If any such techniques are used, they must be noted in the extractions logbook.

7.13 Add a second 60 mL aliquot of methylene chloride to the separatory funnel and extract for the second time (see 7.11 - 7.13). Collect the methylene chloride layer in the same 250 mL amber collection bottle.

7.14 Repeat the extraction for a third time as described in 7.11 - 7.13.

7.15 Proceed to Section 7.30 for extract concentration procedures.

CONTINUOUS LIQUID-LIQUID SAMPLE EXTRACTION (CLLE)

7.16 Set up the CLLE apparatus. All glassware should be rinsed three times with methylene chloride and the extract flasks properly labeled.

7.17 Add 2-3 boiling stones to the round bottom flask and approximately 500 - 600 mL of methylene chloride to the CLLE body.

7.18 Add 1 L laboratory reagent grade water to a CLLE body. This is the method blank for this extraction batch. Be sure that no water leaks into the round bottom flask. A method blank is required for every extraction batch of twenty or fewer samples.

7.19 Measure the initial volume by comparing the meniscus of the sample with the reference bottle of the same bottle type. Please refer to SOP CA-108, "Basic Laboratory Technique", for the reference bottle verification procedure. Record the volume and any notable characteristics (e.g. color, presence of sediment, or odor) in the extraction logbook. Transfer the sample to a CLLE body, being sure that no water leaks into the round bottom flask.

7.20 Prepare an LCS for every daily extraction batch of twenty or fewer samples and each analysis (pesticide and/or PCB). Add 1 L of laboratory reagent grade water to a CLLE body. If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager. When Pesticides and PCBs are extracted together, a LCS and LCSD set must be extracted for each analysis.

7.21 Check the pH of the samples. If it is not between pH 5 and 9, adjust the pH with 10N sodium hydroxide or 1:1 sulfuric acid solution. Note the addition of NaOH or H_2SO_4 in the extraction logbook.

7.22 Transfer two 1 L portions of a sample to CLLE bodies for each analysis for preparation of a matrix spike/matrix spike duplicate if required. An MS/MSD is required if requested by the client or per 20 samples, whichever occurs first. When Pesticides and PCBs are extracted together, a MS and MSD set must be extracted for each analysis. Note: Sufficient sample volume should be available without depleting all remaining sample aliquots. An MS/MSD will be analyzed only if enough sample has been provided by the client. Additionally, in the event that the batch MS/MSD requirement cannot be fulfilled, a laboratory control spike duplicate must be analyzed.

7.23 For each sample, rinse the original sample container with approximately 30 mL of methylene chloride. Add this rinse to the CLLE body.

7.24 Add 1.0 mL of the Pesticide/PCB Surrogate Spike to each sample including the blank, LCS/LCSD and MS/MSD, if performed.

7.25 Add 1.0 mL of Pesticide or PCB Matrix Spike to the appropriate LCS/LCSD and MS/MSD pair, if performed, and stir.

7.26 Attach cooling water Allihn condensers, after first rinsing each 45/50 joint with methylene chloride. Turn on the heating mantles and allow the samples to extract for at least 18 hours, total extract time may go up to 20 hours. Turn off the mantles and let samples cool.

7.27 Proceed to Section 7. 30 for sample extract concentration procedures.

CONCENTRATION OF WATER SAMPLE EXTRACTS

7.28 Rinse the K-D glassware (flask, concentration tube, funnel and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride (or hexane for samples extracted with the Autoextractor) before assembling. Add two boiling chips to the K-D. Insert fluted 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Rinse the sodium sulfate in the assembled funnels with ~20-30 mLs of methylene chloride (hexane for samples extracted with the Autoextractor). Place the assembled K-D's under the funnels.

7.29 For methylene chloride extracts, add approximately 50 mL Hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only.

Note: For Pesticide / PCB samples originating from South Carolina (see worknotes) do not add the hexane at this step. Solvent exchange will be during the nitrogen blow down procedure.

7.30 Transfer the methylene chloride or hexane extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract bottle three times with ~ 2 – 3 mLs of methylene chloride (or hexane for samples extracted with the Autoextractor). Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride (or hexane for samples extracted with the Autoextractor) and allow to drain.

7.31 Transfer the labels from the collection bottles or round bottom flasks (from the CLLE extraction) to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride (or hexane for samples extracted with the Autoextractor).

7.32 Place the K-D in a hot water bath (85-90°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 5-6 mL, remove the K-D from the water bath. Allow the

K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of hexane (methylene chloride for samples that have not gone through solvent exchange (ie. South Carolina samples). Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL hexane.

7.33 Reduce the extracts to ≈ 1 mL using Nitrogen blow-down apparatus. The bath temperature must be no higher than the boiling point of the solvent (45 °C for hexane). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least once or twice with ≈1 mL of hexane (methylene chloride for samples not yet solvent exchanged). The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging needle closer to the surface of the extract to expedite the concentration. Note any problems or extract losses, if they occur, in the extractions logbook. Transfer extract to a 12 or 16 mL vial. Using a reference vial for volume comparison, adjust the final extract volume to 10 mL by rinsing sides of tube with hexane and transferring rinsings to vial.

7.34 For samples that still need to be solvent exchanged, reduce the methylene chloride extract to ~ 1 mL. Add 10 mL of hexane to the concentrator tube and reduce to ~ 1 mL again on the N-evap. Adjust final extract to 10 mL by rinsing sides of tube with hexane and transferring rinsings to vial.

7.35 If at any point in the concentration procedure the concentrator tube goes dry – reextract the sample immediately.

7.36 Transfer the label from the concentrator tube to the vial. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and “tray location” of the individual extract vials.

7.37 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis. All sample extracts for 8081 pesticide analysis do not undergo further cleanup unless requested by the client. Therefore, all sample extracts for combined 8081/8082 analyses must be split. Prior to splitting, mix contents of vial well. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

A method blank must be extracted for each and every item listed below:

- Each day of extraction (24 hours midnight - midnight)
- Each extraction method
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each extraction method
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticide and/or PCB) to be performed

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Quantitation (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of the analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste - Physical/Chemical Methods, Methods 3510C and 3520C, USEPA SW-846, Third Edition, Final Update III, December 1996.

40 CFR 136, Appendix A, "Test Procedures for Analysis of Organic Pollutants," Method 608, June, 1998 edition.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Current Version.

LIST OF TABLES AND FIGURES

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

TABLE 1

SUMMARY OF METHOD MODIFICATIONS (METHOD 3510, current revision)

TOPIC	KATAHDIN SOP CA-515-09	METHOD 3510, current revision
Apparatus/Materials	1. 12 or 16 mL vials used for final extract 2. 250 mL amber bottle or flask used 3. 1.0 mL syringe 4. short stem funnels	1. 2 mL vials used for final extract 2. 250 mL Erlenmeyer flask 3. 5.0 mL syringe 4. drying column
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to separatory funnel	1. one liter graduated cylinders used to transfer initial sample volume to separatory funnel
Procedures	1. extract collection in amber bottle or Erlenmeyer flask 2. extract dried using Na_2SO_4 in short stem funnels 3. no apparatus height specification for concentration on water bath 4. sample removed from water bath when volume reaches ~10 mL 5. hexane added directly to K-D body at start of concentration process (this modification is not allowed for samples originating from South Carolina).	1. extract collection in Erlenmeyer flask 2. extract dried using Na_2SO_4 in drying columns 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. sample removed from water bath when volume reaches 1-2 mL 5. solvent exchange via large K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

TABLE 2

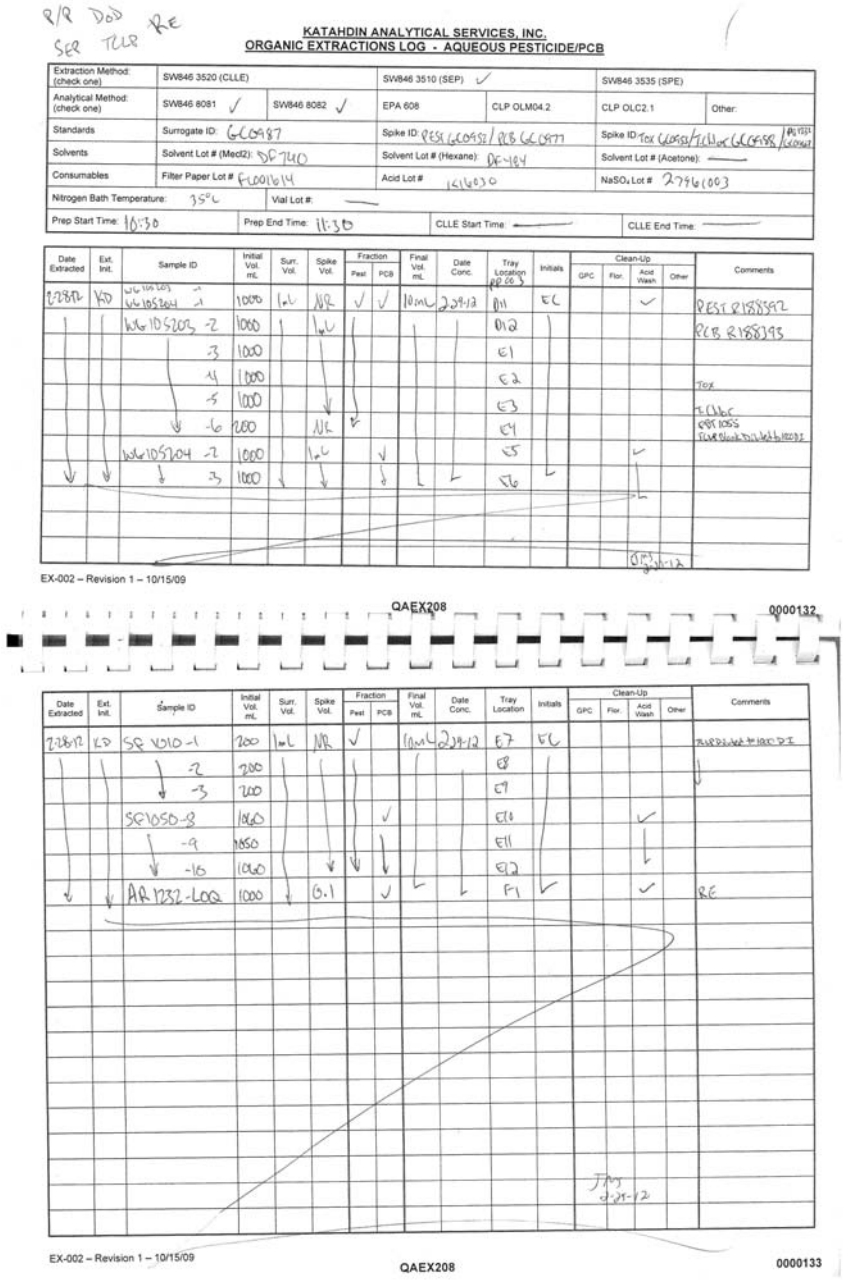
SUMMARY OF METHOD MODIFICATIONS (METHOD 3520, current revision)

TOPIC	KATAHDIN SOP CA-515-09	METHOD 3520, current revision
Apparatus/Materials	1. short-stem funnels 2. 12 or 16 mL vials used for final extract	1. drying columns 2. 2 mL vials used for final extract
Reagents		
Sample preservation/ handling	1. entire contents of 1 L sample bottle transferred to CLLE	1. one liter graduated cylinders used to tranfer initial sample volume to CLLE
Procedures	1. CLLE for 18 $\pm$ 2 hours 2. extract dried using Na_2SO_4 in short stem funnels 3. no apparatus height specification for concentration on water bath 4. sample removed from water bath when volume reaches ~10 mL 5. hexane added directly to K-D body at start of concentration process (this modification is not allowed for samples originating from South Carolina).	1. CLLE for 18-24 hours 2. extract dried using Na_2SO_4 in drying columns 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. sample removed from water bath when volume reaches 1-2 mL 5. solvent exchange via macro K-D with addition of 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: PREPARATION OF AQUEOUS SAMPLES FOR PESTICIDES/PCBs ANALYSIS

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



R/R DOD
SER TCLP RE

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - AQUEOUS PESTICIDE/PCB

Extraction Method: (check one)	SW846 3520 (CLLE)		SW846 3510 (SEP) ✓		SW846 3535 (SPE)
Analytical Method: (check one)	SW846 8081 ✓	SW846 8082 ✓	EPA 608	CLP OLM04.2	CLP OLC2.1	Other:
Standards	Surrogate ID: GC0987		Spike ID: PEST GC0955 / PCB GC0977		Spike ID: Tox GC0955/TCLP Tox GC0955 / GC0977
Solvents	Solvent Lot # (MeCl2): DF740		Solvent Lot # (Hexane): DF-464		Solvent Lot # (Acetone): —
Consumables	Filter Paper Lot # FL001614		Acid Lot # 151603 0		$NaSO_4$ Lot # 2796003
Nitrogen Bath Temperature: 35°C		Vial Lot #: —			
Prep Start Time: 10:30		Prep End Time: 11:30	CLLE Start Time: —		CLLE End Time: —

Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction: Pest	Fraction: PCB	Final Vol. mL	Date Conc.	Tray Location	Initials	Clean-Up: GPC	Clean-Up: Flor.	Clean-Up: Acid Wash	Clean-Up: Other	Comments
7-28-12	KD	WG105204-1	1000	1mL	NR	✓	✓	10mL	7-29-12	D11	EC			✓		PEST R1888592
		WG105203-2	1000	1	1mL					D12						PCB R1888393
		-3	1000							E1						
		-4	1000							E2						Tox
		-5	1000							E3						TCLP
		-6	200		NR					E4						GPC 1055
		WG105204-2	1000		1mL		✓			E5				✓		
		-3	1000							E6						

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Date Extracted	Ext. Init.	Sample ID	Initial Vol. mL	Surr. Vol.	Spike Vol.	Fraction: Pest	Fraction: PCB	Final Vol. mL	Date Conc.	Tray Location	Initials	Clean-Up: GPC	Clean-Up: Flor.	Clean-Up: Acid Wash	Clean-Up: Other	Comments
7-28-12	KD	SE V010-1	200	1mL	NR	✓		10mL	7-29-12	E7	EC					TCLP SW846 1311 DI
		-2	200							E8						
		-3	200							E9						
		SE1050-8	1960				✓			E10				✓		
		-9	1950							E11						
		-10	1960		↓	↓	↓			E12						
↓	↓	AR1232-LOQ	1000	↓	0.1		✓	↓	↓	F1	✓			✓		RE

EX-002 – Revision 1 – 10/15/09

QAEX208 0000133

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-524
Revision History
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TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS

Prepared By: Mike Thomas Date: 7/98

Approved By:

Group Supervisor: Michael F. Thomas Date: 11/15/00

Operations Manager: [signature] Date: 11/15/00

QA Officer: Deborah J. Nadeau Date: 11.15.00

General Manager: [signature] Date: 11/16/00

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Minor changes throughout. Clarifications to procedure section.	DN	11.15.00	11/13/00
02	Added definitions to section 1.1. Wording changed or added to clarify sections 5, 6, 8, + 9. New figure	MRC	11.08.04	11.08.04
03	Sect. 7.1.2 - adding the step to rinse forceps also. 7.10 adding condenser temperature and output voltage of variable transformer	LAD	04/06	04/06
04	Added generated waste information. Updated spike list. Added LCSD. Reworded Sect. 7.10 and 7.11 for clarification. Updated Table 1 Replaced Figure 1	LAD	09/07	09/07
05	Changed "N-Lo" waste to "K" waste. Updated Logbook example. Sect. 7 - added wording instructing the recording of consumable lot #'s in Logbook.	LAD	07/08	07/08

TITLE: PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Added balance criteria. Changed weight criteria to +/- 0.05g. Minor changes to section 7 to reflect current techniques. Clarified samples being GPC'd are not solvent exchanged into hexane. Updated logbook page. Added CA-108 reference for subsampling information	LAD	08/09	08/09
07	Removed targeting sample weights. Removed decanting samples prior to extraction.	LAD	08/10	08/10
08	Minor changes to section 7 to reflect current practices. Updated section 7.6 for frequency of MS/D's. Added information for MDL, LOD and LOQ to section 9. Updated references. Added H_2SO_4 to reagents.	LAD	04/12	04/12

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-524-08**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-524-08**, titled **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS.**

Recipient: _____ Date: _____

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure for extracting pesticides/PCBs from solids such as soils, sludges, and wastes using Method 3540. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent.

This method is applicable to the isolation and concentration of water-insoluble and slightly water-soluble organics in preparation for a variety of chromatographic procedures including methods 8081 for pesticides and 8082 for PCB's.

1.1 Definitions

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, reagent water is used as a blank matrix; for soil samples, baked organic-free sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source, and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, standards, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the extraction of samples for pesticide/PCB analysis. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training and Documentation of Capability".

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

It is the responsibility of all Katahdin technical personnel involved in the extraction of samples for pesticide/PCB analysis to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to indicate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Waste Disposal

Wastes generated during the preparation of samples must be disposed of in accordance with the procedures described in the current revision of the Katahdin Hazardous Waste Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

Any methylene chloride solvent waste generated during the rinsing of glassware etc. should be disposed of in the “D” waste stream satellite accumulation area nearest the point of generation. Acetone and hexane are considered flammable waste, and should be disposed of in the “O” waste stream satellite accumulation area nearest the point of generation. Post-extraction soil samples, used glass wool, and sodium sulfate waste should be disposed of in the soil with organics “I” waste stream

satellite accumulation area nearest the point of generation. Acid waste generated during the cleanup of PCB samples should be disposed of in the "K" satellite accumulation area nearest the point of generation. Please refer to the current revision of SOP CA-107 for the location of satellite waste accumulation areas.

2.0 SUMMARY OF METHOD

2.1 The solid sample is mixed with anhydrous sodium sulfate, placed in a Soxhlet extractor and extracted with methylene chloride.

2.2 The extract is then dried, concentrated, and exchanged into hexane for GC analysis. Sulfuric acid cleanup is performed on extracts for 8082 PCB analysis.

3.0 INTERFERENCES

Solvents, reagents, glassware, and other sample preparation apparatus may yield interferences to GC analysis due to the presence of contaminants. These contaminants can lead to discrete artifacts or elevated baselines in chromatograms. Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks. Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates that are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory. At no time may gloves that have not been tested for phthalates or gloves known to contain phthalates be used or stored in the organic extraction lab. Additionally, whenever possible plastic items in this lab must be replaced with metal or teflon or other non-phthalate plastic substitute.

Special care should be taken to ensure clean glassware and apparatus are used, pre-rinsed with the appropriate solvent prior to use. Solvents should be analyzed prior to use to demonstrate that each lot is free of contaminants that may interfere with the analysis.

Interferences coextracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to inteferences, further cleanup of the sample extract may be needed to minimize interferences.

4.0 APPARATUS AND MATERIALS

4.1 Soxhlet extractor – 45/50 top joint and 24/40 lower joint.

4.1.1 500 mL flat-bottom boiling flask

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4.1.2 Allihn cooling water condenser

4.2 Powder Funnels – 100 mm top diameter, 35 mm stem

4.3 Kuderna-Danish (K-D) apparatus

4.3.1 Concentrator tube - 10-mL

4.3.2 Evaporation flask - 500-mL

4.3.3 Snyder column - Three-ball macro

4.4 Nitrogen evaporation (N-EVAP) apparatus.

4.5 Boiling stones, 12 mesh silicon carbide (carborundum) – pre-purified by Soxhlet extraction in methylene chloride

4.6 Water bath - Heated, with concentric ring cover, capable of temperature control (± 5°C). The bath should be used in a hood.

4.7 Vials - Glass, 4, 12, or 16 mL with Teflon-lined screw caps

4.8 Glass wool (fiberglass) - baked at 400°C for a minimum of 4 hours or overnight.

4.9 Heating mantles - Rheostat controlled.

4.10 Disposable glass Pasteur pipets, 5 ¾", and bulbs.

4.11 Drying oven - capable of maintaining 105°C for glassware drying.

4.12 Muffle oven – capable of maintaining 400 °C for baking glass wool and organic-free sand.

4.13 Beakers, 250 or 400 mL

4.14 Top-loading balance - capable of weighing to 0.**01** g.

4.15 Spatulas, stainless-steel

4.16 Long forceps, stainless-steel

4.17 Metal clips – for securing Soxhlets to boiling flasks

4.18 Filter paper, 18.5 cm, Fisherbrand or Whatman 114V (or equivalent)

4.19 Gel Permeation Chromatograph (GPC) - J2 Scientific AccuPrep MPS^{TM} with internal UV detection

5.0 REAGENTS

5.1 Sodium sulfate (granular, anhydrous and powdered, anhydrous) (ACS reagent grade), Na_2SO_4. Certified by the manufacturer/vendor as purified by heating at 400°C for 4 hours prior to receipt by the laboratory.

5.2 Sulfuric acid solution (1:1 H2SO4 : H2O) – Prepared in an icebath by slowly adding a volume of concentrated H2SO4 to an equivalent volume of reagent water and swirl gently to mix. Caution should be taken when adding the acid to the water as the reaction is highly exothermic.

5.3 Methylene chloride - (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS analysis.

5.4 Acetone and hexane – (pesticide grade or equivalent) purchased by lot, evaluated prior to use by concentration of 300 mL to 1 mL followed by GC/MS and GC analysis.

5.5 Organic-free sand, purified by baking at 400 °C at a minimum of 4 hours or overnight. Method blanks serve as checks on the baked sand.

5.6 Surrogate spiking solution - Prepare a solution of decachlorobiphenyl (DCB) and tetrachloro-meta-xylene (TCMX) at a concentration of 1 ug/mL in acetone.

5.7 Matrix Spike/Lab Control Sample spiking solution

5.6.1 Pesticide spike solution – prepare in pesticide grade methanol containing the analytes listed below at concentrations of 0.5 ug/mL.

4,4'-DDD	Endrin
4,4'-DDE	Endrin Aldehyde
4,4'-DDT	Endrin Ketone
Aldrin	gamma-BHC (Lindane)
alpha-BHC	Heptachlor
beta-BHC	Heptachlor Epoxide
delta-BHC	Methoxychlor
Dieldrin	alpha-Chlordane
Endosulfan I	gamma-chlordane
Endosulfan II	Endrin
Endosulfan Sulfate	Endrin Aldehyde

5.6.2 PCB spike solution – prepare Aroclor 1660 (Aroclor 1016 and 1260) in pesticide grade acetone at a concentration of 5.0 ug/mL each.

5.7 Store the solutions mentioned in sections 5.5 and 5.6 at -10 to -20 °C (±2 °C) in a Teflon sealed container. Solution must be verified by GC/ECD prior to use and must be replaced every 6 months or sooner if degradation is evident.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Sediment/soil samples must be collected in a soil jar and must be maintained at 4°C (±2°C).

Holding time for extraction of sediment/soil samples for Method 3540 is 14 days from date of sample collection, although the analyst should be aware that actual holding times employed may be project/program specific.

Store all extracts at 4°C (±2°C) in the dark in labeled Teflon-sealed containers. See SOP SD-902, "Sample Receipt and Internal Control," current revision, for storage areas and temperature maintenance procedures.

7.0 PROCEDURES

The following information must be recorded in the extraction logbook.

- Extraction method
- Surrogate and spike IDs
- Lot numbers of all solvents, acids and bases, sodium sulfate, filter paper
- Nitrogen evaporation water bath temperature
- pH if applicable
- Extraction and Concentration dates
- Extraction and Concentration analyst
- Soxhlett extraction start and end times, also the prep start and end times
- Sample ID or QC sample ID
- Initial and final volumes or weight
- Surrogate and spike amounts
- Final extract tray location
- Any comments regarding the sample extraction (ie. Emulsion)

Samples need to be “swiped” out when removing and “swiped” in when replacing samples in storage locations to maintain the internal chain of custody. Refer to Katahdin SOP, Sample Receipt and Internal Control, current revision, for the proper procedure for removal and return samples.

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7.1 Preparing the Soxhlet Extraction Apparatus

7.1.1 Rinse the Soxhlet extractors and 500 mL flat-bottom boiling flasks three times with methylene chloride. Be sure that the solvent rinses through the large vapor tube and smaller siphon tubes of the Soxhlet. Inspect these for tiny cracks. Also rinse the 24/40 lower joint.

7.1.2 Add ~ 250 mLs of methylene chloride to the 500 mL boiling flask. Add several boiling stones. Rinse the stainless steel forceps with Methylene chloride. Working in a hood, place a plug of the glass wool at the bottom of the Soxhlet so that the siphon tube hole is covered. Insert the 24/40 joint of the Soxhlet extractor into the 500 mL boiling flask and secure with a metal clip. Cover the top of the Soxhlet extractor with a piece of aluminum foil until ready to begin loading the sample.

7.2 Sample Handling

7.2.1 Sediment/soil samples – Do not decant any water layer on a sediment sample. Mix the sample thoroughly with the stainless steel spatula. If the sample container is full to the extent that stirring the sample is impractical, try to remove the “best representative” aliquot from the jar based on color, particle size, moisture, etc. Discard any foreign objects such as sticks, leaves, and rocks.

7.2.2 Gummy, fibrous, or oily materials not amenable to mixing should be cut, shredded, or otherwise reduced in size to allow for maximum exposure of the sample surfaces to the extraction solvent. Materials such as glass, rubber, metal, etc. may not require mixing with powdered sodium sulfate to disperse the sample. Plastic materials must be tested for degradation (melting) in methylene chloride prior to Soxhlet extraction.

7.2.3 Please refer to Katahdin Analytical Services SOP CA-108, current revision, “Basic Laboratory Techniques” for more information of subsampling.

7.3 Weigh out an approximate, greater than 30 g portion of sample into a labeled 400 mL beaker. Record sample weight to nearest 0.01 g in appropriate extraction logbook. Add between 30 to 60 g of powdered sodium sulfate, as required, to produce a “free-flowing” mixture. The amount of sodium sulfate added will depend upon the moisture content of the sample (e.g., low moisture content will require less sodium sulfate). Mix well with a spatula. Keep the spatula in the sample beaker and cover the beaker with aluminum foil. Record sodium sulfate lot in logbook.

7.4 A method blank must be prepared with each extraction batch, not to exceed 20 client samples. To prepare method blank, weigh out one greater than 30 g portion of purified

sand in a labeled 400 mL beaker. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Add 60 g sodium sulfate and mix well. (Although a "free-flowing" mixture can be achieved with less than 60 g sodium sulfate, the method blank must contain 60 g in order to evaluate the sodium sulfate as a potential source of contamination.)

7.5 A laboratory control sample (LCS) must be prepared with each extraction batch, not to exceed 20 client samples. To prepare LCS, weigh out one greater than 30 g portion of purified sand in a labeled 400 mL beaker. Record sample weights to nearest 0.01 g in appropriate extraction logbook. Add 30 g sodium sulfate and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB LCS's must be prepared (refer to section 5.6). If an MS/MSD pair is not extracted on a particular day, an LCS/LCSD pair may be required in order to meet client-specific or program-specific requirements. This information will be disseminated from the project manager or Department Manager.

7.6 A matrix spike/matrix spike duplicate (MS/MSD) set should be prepared for every 20 samples. An MS/MSD will be analyzed only if enough sample has been provided by the client. Additionally, in the event that the batch MS/MSD requirement cannot be fulfilled, a laboratory control spike duplicate must be analyzed. To prepare MS/MSD, weigh out two approximate, greater than 30g portions of the sample designated for MS/MSD into each of two labeled 400 mL beakers. Record sample weights to nearest 0.01g in appropriate extraction logbook. Add 30 g sodium sulfate to each to produce a free-flowing mixture, and mix well. With extraction batches prepared for combined 8081/8082 Pesticide and PCB analysis, separate Pesticide and PCB MS/MSD pairs must be prepared (refer to section 5.6).

7.7 Once all of the QC and field samples have been weighed and mixed with sodium sulfate, begin adding each to the assembled and appropriately labeled Soxhlet extractors using the stainless steel spatulas. Carefully scrape all of the mixtures from the beaker walls so that no more than 1% remains behind in the beaker. Be careful that none of solid material falls into the extract flask through the large vapor tube.

7.8 To all samples, method blank, LCS/LCSD, and MS/MSD add 1.0 mL (if FV=10mL, adjust amount for different final volumes) of the pesticide/PCBs surrogate spiking solution using a 1.0 mL gas tight syringe. Record surrogate spike volume and identification code in the extraction logbook. Thoroughly rinse syringe with solvent between each use.

7.9 To LCS/LCSD and MS/MSD add 1.0 mL (if FV=10mL, adjust amount for different final volumes) of either the pesticide or PCBs matrix spike/LCS spiking solutions using a 1.0 mL gas tight syringe. Record matrix spike/LCS spiking solution volume

and identification codes in the extraction logbook. Thoroughly rinse syringe with solvent between each use.

7.10 Rinse the joints of the Allihn cooling condensers with Methylene Chloride, collecting the waste in a methylene chloride solvent waste container. Place each of the Soxhlet extractors in a heating mantle and lower the Allihn cooling water condensers into the 45/50 joints of the extractors. The condensers should be set to a temperature of 15ºC. Save the pieces of aluminum foil for covering the Soxhlets when the extraction is complete. Switch on the individual heating mantles and be sure that the Rheostat of the variable transformer is set to 55% of the output voltage. Once the methylene chloride begins to boil and the Soxhlet begins to cycle (solvent will immerse the sample and collect in the Soxhlet until the level reaches that of the small siphon tube and then begin to spill over into the extract flask), re-check the apparatus' for leaks. Allow the samples to extract for 18-24 hours. Be sure the chiller/recirculator temperature is set low enough to provide enough cooling capacity for the number of extractions in the batch.

7.11 When the extraction is complete, allow the extracts to cool before dismantling. Remove the Allihn condenser and replace the aluminum foil on top of the extractor. Move the extractors to a hood and detach the extractor from the extract flask. Tilt each extractor slightly to cause any remaining solvent in the sample chamber to drain through the siphon tube into the extract flask. This will help to cool the extract flask and make the apparatus easier to dismantle. Try to drain as much solvent as possible from the extractor into the flask. **This is done by rinsing a glass tube in methylene chloride and pressing on the sample slightly so that as solvent as possible is drained into the extract flask.** Cover the flask with aluminum foil and store in the interim extract storage refrigerator unless the extracts are to be concentrated the same day.

7.12 Immediately remove the extracted soil/sodium sulfate mixtures from the extractors using a square edge spatula, and dispose of in an appropriate solid waste container. It is important to do this soon after the extractors are dismantled, as the sample mixture will tend to "freeze" into a solid mass in the Soxhlet as the solvent dries.

CONCENTRATION OF THE EXTRACTS

7.13 Rinse the K-D glassware (flask, concentration tube, and Snyder column, including the ground glass joints on the flask and columns) three times with methylene chloride before assembling. Add a few boiling chips to the K-D. Insert 18.5 cm filter papers into short stem powder funnels and add ~ 2 inches of sodium sulfate crystals. Place the assembled K-D's under the funnels. Record the lot numbers of the solvent, sodium sulfate and filter papers in the extraction logbook.

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

7.14 If samples are to be GPC'd, refer to the current revision of Katahdin SOP CA-513, Extract Cleanup Using Gel Permeation Chromatography, for appropriate concentrating procedures. Samples that undergo GPC are not solvent exchanged into hexane. All pesticide soil samples should be cleaned up to reduce matrix interferences.

7.15 If samples are not to be GPC'd follow Steps 7.16 through 7.23 to concentrate extracts to final volume of 10 mLs (or a client specified final volume)

7.16 For a solvent exchange, (for samples not being GPC'd), add approximately 50 mL hexane to funnel and let drain through. Since methylene chloride has a lower boiling point than Hexane, this will result in a final extract in hexane only. Record the lot number of the solvent in the extraction logbook.

7.17 Transfer the extracts to the K-D concentrator setups through the sodium sulfate in the funnels. This is the drying step, which is required to remove residual water from the extracts. Any large water layers must be removed by other means, prior to pouring through the sodium sulfate. After pouring all of the extract volume through the sodium sulfate, rinse the extract flask three times with ~ 2 – 3 mLs of methylene chloride. Add the rinsings through the sodium sulfate to complete a quantitative transfer. Rinse the sodium sulfate with ~ 15 mLs of methylene chloride and allow to drain.

7.18 Transfer the labels from the extract flasks to the K-Ds. Remove the funnel and attach a 3-ball macro Snyder column. Pre-wet the Snyder column with 1 mL of methylene chloride.

7.19 Place the K-D in a hot water bath (75-85°C). Gently swirl K-D in the water until boiling begins. At the proper distillation rate, the Snyder balls should chatter but the chambers should not flood with condensed solvent. The K-D should be kept in a vertical orientation while on the bath. When the apparent volume in the concentrator tube reaches ≈ 6 mL, remove the K-D from the water bath. Allow the K-D to cool for 10 minutes. Rinse the Snyder column lower joint with ≈ 1 mL of methylene chloride, hexane, if exchange is taking place. Remove the Snyder column. Wipe off any water from the neck above the lower joint of the flask. Separate the K-D flask from the concentrator tube, rinsing the ground glass joint with ≈ 1 mL methylene chloride, hexane, if exchange is taking place.

7.20 Reduce the extract in the concentrator tube to approximately 1-2 mL using the nitrogen blow-down apparatus to ensure all methylene chloride has been evaporated. The bath temperature must be no higher than the boiling point of the solvent (39°C for methylene chloride). Turn the gas to 3 psi. Be careful not to splash the extract out of the tube. <u>During concentration on the N-evap, the internal wall of the concentrator tube and the N-evap sparging pipet must be rinsed down at least</u>

once or twice with ≈1 mL of hexane. The solvent level in the concentrator tube must be positioned below the level of the water bath as much as possible to prevent water from condensing into the sample extract. As the extract volume is reduced, lower the N_2 sparging pipet closer to the surface of the extract to expedite the concentration. Record the temperature of the water in the nitrogen evaporation water bath in the extraction logbook, also note any problems or extract losses, if they occur.

7.21 Complete quantitative transfer of the extract to a vial by using hexane. Adjust the volume of the hexane extract to 10 mL (or a client specified final volume) in either a 12 or 16 mL vial using the appropriate "reference vial" for volume comparison.

7.22 Label the vial with lab sample number, extraction date, matrix and analysis. Store extract vials at a temperature of 4 ± 2 °C until ready for analysis. Indicate in the extractions logbook the box number and "tray location" of the individual extract vials.

7.23 All sample extracts for 8082 PCB analysis must undergo a sulfuric acid wash (cleanup) prior to analysis, unless it has been GPC'd. All sample extracts for 8081 pesticide analysis should undergo further cleanup using the GPC unless time is a factor. All sample extracts for combined 8081/8082 analyses must be split unless GPC'd. One portion must be acid cleaned for 8082 analysis. The associated method blank must be split and acid-cleaned in the same fashion. PCB LCSs and matrix spikes are acid cleaned also. Pesticide LCSs and matrix spikes are not subjected to further cleanup. Record the lot number of the acid in the extraction logbook. Please refer to Katahdin SOP CA525 (current revision), Extract Cleanup Using Sulfuric Acid, for further instructions.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

A method blank must be extracted for each and every item listed below:

- Each sample matrix (soil, water)
- Each day of extraction (24 hours midnight - midnight)
- Each extraction method or level
- Every 20 samples extracted in a 24-hour period

A laboratory control sample (LCS) is required for each and every item listed below:

- Each sample matrix
- Each extraction method or level
- Every extraction batch of twenty or fewer samples
- Each analysis (pesticide and/or PCB) to be performed

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

Refer to the current revision of the applicable Katahdin SOP for analysis of Pesticides and PCBs for quality control acceptance criteria.

Each extractions analyst must demonstrate proficiency in performing the extractions that prepare samples for analysis. Demonstration consists of preparation (extraction), by the analyst, of at least four aliquots which are then analyzed according to the analytical method in question. These QC samples must meet all quality control acceptance limits. Demonstration must be documented by use of a form which summarizes the results of the analysis of these aliquots, calculated percent recoveries, and standard deviation. Demonstration of proficiency must be done one time per analyst initially and then annually thereafter. Refer to SOPs QA-805 and QA-807, current revision.

If, upon analysis of the extracted samples, it is discovered that quality control acceptance criteria have not been met, all associated samples must be evaluated against all the QC. In some cases data may be reported, perhaps with narration, while in other cases, other corrective action may be taken. The corrective actions may include re-extraction of the samples associated with the quality control sample that did not meet acceptance criteria, or may include making new reagents and standards if the standardization is suspect. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory

is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Detection (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the applicable analytical SOP for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste-Physical/Chemical Methods, Method 3540C, SW-846, Third Edition, Updates I, II, IIA, IIB, and III Revised December 1996, US EPA.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Current Version.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010..

LIST OF TABLES AND FIGURES

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

TABLE 1

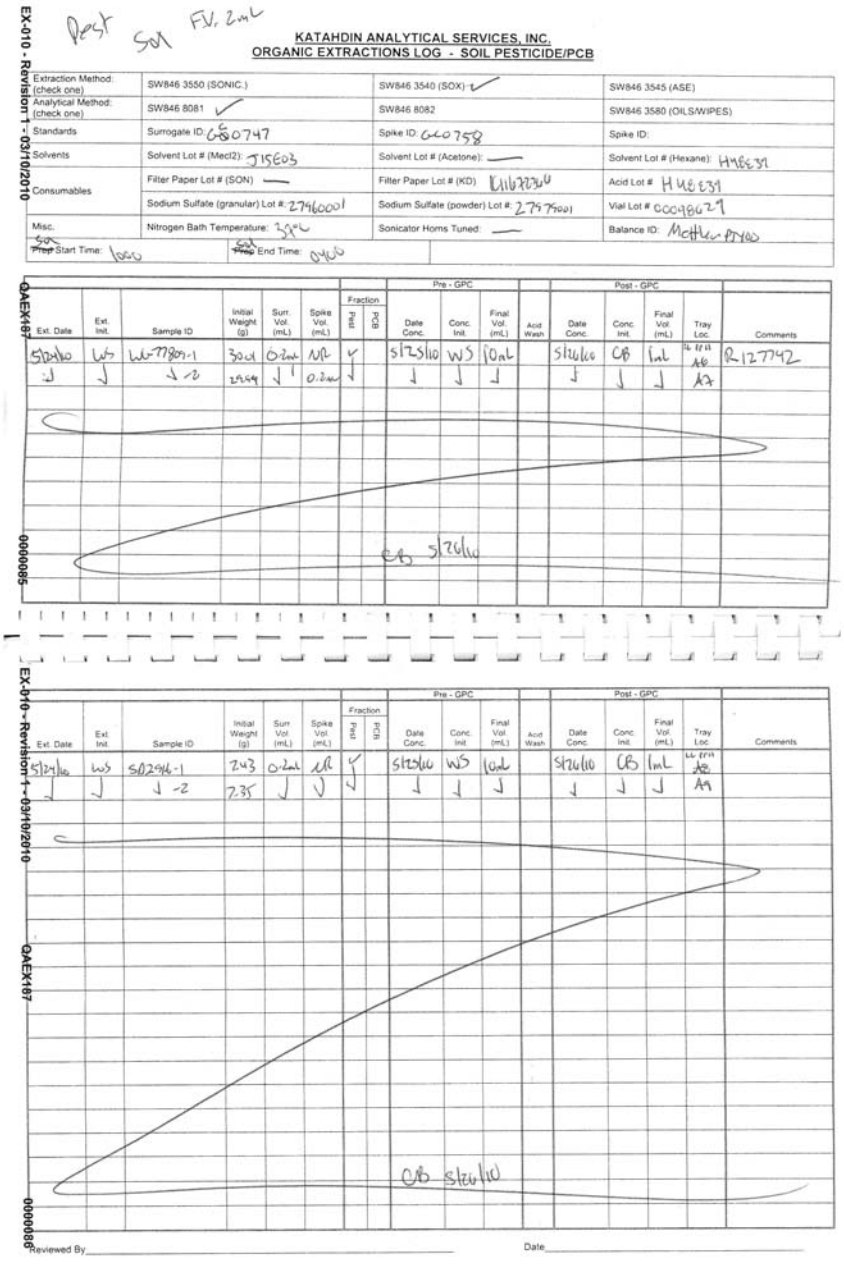
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-524-08	METHOD 3540, current revision
Apparatus/Materials	1. short stem funnels	1. drying columns
Reagents		
Sample preservation/ handling		
Procedures	1. Use 30 grams of sample and 30 grams of sodium sulfate. 2. Use 250 mL of methylene chloride 3. no apparatus height specification for concentration on water bath 4. water bath at 75-85 deg C 5. sample removed from water bath when volume reaches ~6 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane at the start of concentration process	1. Use 10 grams of sample and 10 grams of sodium sulfate. 2. Use 300 mL of methylene chloride 3. partially immerse concentrator tube in water and lower apparatus to complete concentration in 10-15min 4. water bath at 80-90 deg C 5. sample removed from water bath when volume reaches 1-2 mL 6. Solvent exchange to hexane is performed using K-D apparatus with addition of approximately 50 mL hexane after concentrating methylene chloride extract to 1 mL
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		

TITLE: **PREPARATION OF SEDIMENT/SOIL SAMPLES BY SOXHLET EXTRACTION USING METHOD 3540 FOR PESTICIDE/PCB ANALYSIS**

FIGURE 1

EXAMPLE OF LOGBOOK PAGE



Pest Soil FV. 2mL

KATAHDIN ANALYTICAL SERVICES, INC.
ORGANIC EXTRACTIONS LOG - SOIL PESTICIDE/PCB

Extraction Method: (check one)	SW846 3550 (SONIC.)	SW846 3540 (SOX) ✓	SW846 3545 (ASE)
Analytical Method: (check one)	SW846 8081 ✓	SW846 8082	SW846 3580 (OILS/WIPES)
Standards	Surrogate ID: GS0747	Spike ID: GC0758	Spike ID:
Solvents	Solvent Lot # (MeCl2): J15E03	Solvent Lot # (Acetone): —	Solvent Lot # (Hexane): HXBE31
Consumables	Filter Paper Lot # (SON) —	Filter Paper Lot # (KD) K1167736	Acid Lot # HXBE31
	Sodium Sulfate (granular) Lot #: 2746001	Sodium Sulfate (powder) Lot #: 2747501	Vial Lot # C004986Z9
Misc.	Nitrogen Bath Temperature: 35°C	Sonicator Horns Tuned: —	Balance ID: Mettler AT400
~~Prep~~ Start Time: 1000	~~Prep~~ End Time: 0400		

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction: Pest	Fraction: PCB	Pre-GPC: Date Conc.	Pre-GPC: Conc. Init.	Pre-GPC: Final Vol. (mL)	Acid Wash	Post-GPC: Date Conc.	Post-GPC: Conc. Init.	Post-GPC: Final Vol. (mL)	Post-GPC: Tray Loc.	Comments
5/24/10	WS	WS-77809-1	30.0	0.2mL	NA	✓		5/25/10	WS	10mL		5/26/10	CB	1mL	A6	R127742
↓	↓	↓ -2	29.9	↓	0.2mL	↓		↓	↓	↓		↓	↓	↓	A7	

CB 5/26/10

Ext. Date	Ext. Init.	Sample ID	Initial Weight (g)	Surr. Vol. (mL)	Spike Vol. (mL)	Fraction: Pest	Fraction: PCB	Pre-GPC: Date Conc.	Pre-GPC: Conc. Init.	Pre-GPC: Final Vol. (mL)	Acid Wash	Post-GPC: Date Conc.	Post-GPC: Conc. Init.	Post-GPC: Final Vol. (mL)	Post-GPC: Tray Loc.	Comments
5/24/10	WS	SO2946-1	7.43	0.2mL	NA	✓		5/25/10	WS	10mL		5/26/10	CB	1mL	A8	
↓	↓	↓ -2	7.35	↓	↓	↓		↓	↓	↓		↓	↓	↓	A9	

CB 5/26/10

Reviewed By_____ Date_____

EX-010 - Revision 1 - 03/10/2010 QAEX187 0000085 0000086

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Jessica Spearin-Wildes

Review Date: 3-15-13

SOP Number: CA-524

SOP Title: Preparation of sediment/soil samples by Soxhlet extraction using method 3540 for Pest/PCB's

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 3-19-13

QAO Signature: Leslie Dimond **Date:** 032113

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Prepared By: Peter Lemay Date: 4/98

Approved By:

Group Supervisor: Peter Lemay Date: 1/15/01

Operations Manager: John C. Bunton Date: 1/15/01

QA Officer: Deborah J. Nadeau Date: 1.22.01

General Manager: Duncan C. Kilgore Date: 1/16/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 8082	Format changes, added pollution prevention, minor changes to sections 7, 8 and Table 1	DN	1.22.01	1/02/01
02 8082	Revised sections 7.3.1, 7.4.5 and 7.6.1 to be compliant with South Carolina requirements.	DN	5.23.01	5.23.01
03 8082	Changed to practice of reporting higher value. Other minor changes to sections 7.5.2, 7.7.3 + to Table 2.	DN	5.21.02	5.21.02
04 8082	Revised SOP to indicate Turbochrom is being used as instrument control + data collection software. Included Target-related definitions. Changes to sections 7.7.3, 7.7.4 and 7.8.	MRC	08.20.04	08.20.04
05 8082	Changed 7.5.2 to reflect alternating CV Changed Table 2 Sect. 7.3.1 New Checklist added wording to sect. 8	LAD	020305	020305

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06 8082	Changed PCB 1260 to Aroclor 1260. Removed references to 3541. Updated table 2. Added instructions to shake extract before vialing	LAD	04/06	04/06
07	Added waste streams to sect. 1.0. Added ICV to definitions, sect. 5, sect. 7 and Table 1. Added wording regarding 2nd column confirmation criteria and flagging rules to sect. 7.7.4. Added CCV criteria to sect. 7.5.3 and Table 1. Added wording regarding MI to sect. 7.7.3	LAD	08/07	08/07
08	Added tissue, wipe and oil matrices. Added extraction method 3535. Added DDT analog interference, Std. information and analysis frequency criteria. Added HTs are a recommendation. Added Note that 2 detectors must be used for dual column. Updated method references. Removed calibration and surrogate method mod. from Table 2. Added more info @ linear calib. Added extraction references,	LAD	02/09	02/09
09	Added Chemstation to definitions. Clarified that surrogates are added to only the aroclor 1660 standards, not ALL standards.	LAD	05/09	05/09
10	Revised Sections 7, 8, and 10 to reflect compliance with the DoD QSM version 4.1	LAD	08/09	08/09
11	Added Table 2 with DoD QSM Ver. 4.1 QC criteria. Minor changes to Table 1.	LAD	04/10	04/10
12	Removed Sect. 4.5 - Analytical balance. Removed Sect. 5.2.4 - DDT analog standard. Removed Sect. 7.5 - DDT analog standard analysis requirement. Table 1 - Added aver. cal. Criteria and corrected ECV & LCS acceptance criteria. Added and removed references to Sect. 10. Updated Figure 2 - data review checklist. Added PCBs 1262 & 1268	LAD	07/11	07/11
13	Added Extraction method 3546. Removed Quickforms references. Added reporting from KIMS. Updated Figures 1 and 2	LAD	02/13	02/13

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-329-13** titled **ANALYSIS OF AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-329-13** titled **ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082**.

Recipient: _____ Date: _____

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

1.0 SCOPE AND APPLICATION

This SOP describes all aspects of the analysis of extracts of aqueous, solid, tissue, wipe and oil samples for PCBs by EPA Method 8082A as performed by Katahdin Analytical Services, Inc. including sample analysis, data review, standard preparation and instrument calibration.

It is applicable to the following compounds: Aroclor-1016, Aroclor-1221, Aroclor-1232, Aroclor-1242, Aroclor-1248, Aroclor-1254, Aroclor-1260, Aroclor-1262 and Aroclor-1268. Extracts are analyzed by Gas Chromatography-Electron Capture Detector (GC-ECD).

1.1 Definitions

ANALYTICAL BATCH: 20 or fewer samples which are analyzed together with the same method sequence and the same lots of reagents and with the manipulations common to each sample within the same time period or in continuous sequential time periods.

METHOD BLANK (laboratory reagent blank): An artificial sample designed to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus. For aqueous samples, laboratory reagent grade water is used as a blank matrix; for soil samples, muffled sand is used as a blank matrix. The blank is taken through the appropriate steps of the process.

CALIBRATION CHECK: Verification of the ratio of instrument response to analyte amount, a calibration check is done by analyzing for analyte standards in an appropriate solvent. Calibration check solutions are made from a stock solution that is different from the stock used to prepare standards.

CALIBRATION STANDARD (WORKING STANDARD): A solution prepared from the stock standard solution that is used to calibrate the instrument response with respect to analyte concentration.

INDEPENDENT CALIBRATION VERIFICATION STANDARD (ICV): A solution prepared from a stock standard solution independent of the calibration mix that is used to verify the calibration.

LABORATORY CONTROL SAMPLE (LCS): A blank that has been spiked with the analyte(s) from an independent source and is analyzed exactly like a sample. Its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The matrix used should be phase matched with the samples and well characterized.

MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD): Predetermined quantities of stock solutions of certain analytes are added to a sample matrix prior to sample extraction and analysis. Samples are split into duplicates, spiked and analyzed. Percent

recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision.

STANDARD CURVE (CALIBRATION CURVE): A curve that plots concentration of known analyte standard versus the instrument response to the analyte.

STOCK STANDARD SOLUTION: A concentrated solution containing a single certified standard that is a method analyte, or a concentrated solution of a single analyte prepared in the laboratory with an assay reference compound. Stock standard solutions are used to prepare calibration standards.

SURROGATES: Organic compounds which are similar to analytes of interest in chemical composition, extraction and chromatography, but which are not normally found in environmental samples. These compounds are spiked into all blanks, Aroclor 1660 standard, samples and spiked samples prior to analysis. Percent recoveries are calculated for each surrogate.

KATAHDIN INFORMATION MANAGEMENT SYSTEM (KIMS): A complete multi-user system with the capabilities of integrating laboratory instrumentation, generating laboratory worksheets, providing complete Lab Order status and generating reports. KIMS utilizes these features through a database.

PE NELSON TURBOCHROM OR HP CHEMSTATION: data acquisition systems that are used to collect chromatographic data. The systems can also be used to archive raw data files.

TARGET: A software system that combines full processing, reporting and comprehensive review capabilities, regardless of chromatographic vendor and data type.

TARGET DB: An oracle database used to store and organize all Target data files.

1.2 Responsibilities

1.2.1 This method is restricted to use by, or under the supervision of analysts experienced in the analysis of PCBs by method 8082. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, “Personnel Training & Documentation of Capability,” current revision.

1.2.2 It is the responsibility of all Katahdin technical personnel involved in analysis by method 8082 to read and understand this SOP, adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be

recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

1.2.3 It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented and to initiate periodic review of the associated logbooks.

1.3 Health and Safety

1.3.1 Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs (material safety data sheets) for all the materials used in this procedure.

1.3.2 Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

1.4.1 Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Management Program for further details on pollution prevention techniques.

1.4.2 Wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Analytical Environmental Health and Safety Manual and SOP SD-903, “Sample Disposal,” current revision. Expired standards are lab packed, placed in the Katahdin hazardous waste storage area, and disposed of in accordance with this SOP.

1.4.3 Wastes generated during standards preparation are disposed of in the Mixed Flammable Waste (O). After the extracts have been analyzed, the autosampler vials and any expired standard vials or ampules are disposed of in the PCB Vial Waste (H).

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

2.0 SUMMARY OF METHOD

2.1 Method 8082 provides gas chromatographic conditions for the detection of PPB concentrations of certain PCBs. Prior to the use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids (Method 3580, waste dilution) may be analyzed by direct injection. A 2 to 5 ul aliquot of sample is injected into a gas chromatograph (GC) using the direct injection technique, and compounds in the GC effluent are detected by an electron capture detector (ECD).

2.2 The sensitivity of Method 8082 usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, Method 8082 may also be performed on samples that have undergone the following cleanups: Method 3660 - Sulfur Cleanup and Method 3665 - Sulfuric Acid Cleanup.

3.0 INTERFERENCES

Interferences by phthalate esters can pose a problem in PCB determinations when using the electron capture detector. Common flexible plastics contain various amounts of phthalates. Care has to be taken to avoid using any plastic materials during the extraction process. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

Compounds from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides including the DDT series.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph

4.1.1 GC Hewlett Packard 5890 series I or II connected to the Turbochrom or HP Chemstation data system, or equivalent.

4.1.2 Columns - Instruments are configured with a pre-column originating from the injection port, which is connected to a deactivated glass Y splitter that connects two different columns to two detectors. The most commonly used columns are: RTX-35 30M x 0.53 mm ID, RTX-5 30M x 0.53 MM ID, or RTX-1701 30M x 0.53 mm ID. Equivalent columns can be used.

4.1.3 Detectors: Electron capture detectors (ECD). Note: Two detectors must be employed when using dual columns.

4.2 Volumetric flasks, class A: sizes as appropriate with the ground-glass stoppers.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

4.3 Syringes: various sizes for preparing standards and injecting samples on the instrument.

4.4 Vials: various sizes and types including crimp tops.

4.5 Refrigerator for storage of extracts and standards.

5.0 REAGENTS

5.1 Solvents

5.1.1 Hexane: pesticide quality or equivalent for diluting samples and standards.

5.2 Standards

5.2.1 Stock standard solutions: Solutions purchased from suppliers like Restek or other acceptable retailers. Expiration dates are one year from date of opening vial or sooner if manufacturers date is less. Upon receipt, all standards are logged into the appropriate logbook with the date of receipt, expiration date, source, lot number, solvent and concentration of compounds. Standard solutions are stored at 4°C in polytetrafluoroethylene (PTFE)-sealed containers in the dark.

5.2.2 Calibration standards: Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in standards prep logbook. The concentrations of the working PCB calibration standards are 0.05 ug/ml, 0.10 ug/ml, 0.25 ug/ml, 1.0 ug/ml, 2.5 ug/ml, and 10.0 ug/ml. The Aroclor 1660 standard also contain the surrogates Tetrachloro-m-xylene (TCX) and Decachlorobiphenyl (DCB) at the respective concentrations: 0.001 ug/ml, 0.002 ug/ml, 0.005 ug/ml, 0.020 ug/ml, 0.050 ug/ml, and 0.20 ug/ml.

5.2.3 Independent Calibration Verification standard (ICV): Prepared through the dilution of the stock standards with hexane. Expiration date is 6 months or sooner. Information is documented in standards prep logbook. The concentration of the ICV PCB standard is 1.0 ug/ml.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Note: The holding time above is a recommendation. PCBs are very stable in a variety of matrices, and holding times under the conditions listed above may be as long as a year.

7.0 PROCEDURES

7.1 Extraction

Refer to the appropriate SOPs for the correct extraction procedure. In general, water samples are extracted using methods 3510, 3520 or 3535 while solid samples use methods 3540, 3545, 3546 or 3550. Tissue samples are extracted using method 3545 or 3540. Wipes and oils are generally extracted using method 3580.

7.2 Instrument conditions

Refer to the instrument logbook for the current column and conditions.

Typical conditions are:

Makeup flow: 60 ml/min Helium, Ar/Methane or Nitrogen
Column flow: 6 ml/min
Injector Temp: 200
Detector Temp: 300
Oven Ramp: 160(0) - 5/min - 260(10)
Run time: 30 min
Injection size: 2 ul

7.3 Calibration

7.3.1 The GC system is calibrated using the external standard calibration procedure. Six-point calibration standards of Aroclor 1660 (Aroclor 1016 and Aroclor 1260), Aroclor 1242, Aroclor 1248 and Aroclor 1254 are prepared. Six-point calibration standards of Aroclor 1221, Aroclor 1232, Aroclor 1262 and Aroclor 1268 are also prepared. If Aroclor 1221, Aroclor 1232, Aroclor 1262 and Aroclor 1268 are suspected, then a six-point curve of the respective Aroclor will be analyzed prior to the analysis of samples. At a minimum, a single point calibration standard is analyzed for these Aroclors. If using a single point and the Aroclor is required for a project and is detected in a sample, then the GC would be calibrated for the Aroclor and the samples would be reanalyzed.

Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. Three to five characteristic peaks from each Aroclor are used to calibrate a curve. The Target system will calculate a peak height for all three to five peaks in each Aroclor. A separate

calibration curve for each of the three to five peaks can be prepared in Target using the peak height against the concentration of the standard. A non-linear calibration applying a second order polynomial (quadratic fit) equation is used to prepare the curve. In order to be used for quantitative purposes, the Coefficient of Determination (r^2) must be greater than or equal to 0.990. The quadratic equation is:

$$y = ax^2 + bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = the intercept

7.3.2 A non-linear calibration model may not be allowable for certain states, federal programs, or clients. South Carolina does not allow non-linear calibration work originating in their state. In these cases, a linear calibration model must be used. Each calibration standard is injected using the technique that is used to introduce the actual samples into the GC. Three to five characteristic peaks from each Aroclor are used to calibrate a curve. The Target system will calculate a peak height for all three to five peaks in each Aroclor. A separate calibration curve for each of the three to five peaks can be prepared in Target using the peak height against the concentration of the standard.

7.3.2.1 Linear calibration using the average calibration factor

The calibration factor (CF) is calculated using the following formula:

$$CF = A_s / C_s$$

Where: A_s = Peak area (or height) of the analyte or surrogate.
C_s = Concentration of the analyte or surrogate, in µg/L.

To evaluate the linearity of the initial calibration, calculate the mean CF, the standard deviation (SD), and the RSD.

If the RSD of the calibration factor is less than or equal to 20% over the calibration range, then linearity through the origin may be assumed, and the average calibration or response factor may be used to determine sample concentrations.

7.3.2.2 Linear calibration using a least squares regression

$$y = bx + c$$

where: y = Instrument response
b = Slope of the line
x = Concentration of the calibration standard
c = the intercept

The analyst should not force the line through the origin, but have the intercept calculated from the five data points. In addition, do not include the origin (0,0) as a sixth calibration point. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.995. The ICAL must be successful before any samples or other QC check samples can be analyzed.

7.3.3 The AR1660 calibration curve must be checked initially by analyzing a standard containing the same analytes as the curve but prepared from another source. If the response of the analytes from the independent source varies by more than ± 20%, a new independent source standard must be analyzed or a new calibration curve must be prepared and/or analyzed.

7.3.4 The working calibration curve must be verified prior to sample analysis and every 10 samples thereafter by injecting the mid-point calibration standard. If the response for any analyte varies from the expected response by more than ± 15%, a new calibration curve must be prepared for that analyte. The average result for 5 peak heights of the Aroclors is used for quantitation.

For clients or projects requiring DoD QSM 4.1, the response for any analyte must not vary from the expected response by more than ± 20%, or a new calibration curve must be prepared for that analyte. If the CCV fails the above criteria, reanalyze all samples since the last successful calibration verification. If reanalysis cannot be performed, data must be qualified and explained in the narrative. Additionally, apply a Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.

7.4 Retention time windows

7.4.1 Three injections are made of all the PCBs throughout the course of a 72 hour period.

7.4.2 A major peak from the envelope is chosen and a standard deviation is calculated using the three retention times for that peak.

7.4.3 Plus or minus three times the standard deviation of the retention times for each standard is used to define the retention time window; however, the experience of the analyst should weight heavily in the interpretation of chromatograms. The analyst should use the retention time window, but should primarily rely on pattern recognition.

7.4.4 Retention time windows are calculated for each standard on each GC column at method setup and after major maintenance, including whenever a new GC column is installed. The data is kept on file in the laboratory.

7.4.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.07 for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.

7.5 Gas chromatographic analysis

7.5.1 Shake samples and let them sit for one minute before vialing for analysis.

7.5.2 All instrument injections are performed using the direct injection technique with an autosampler set for 2-5 ul injection volumes.

7.5.3 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration as listed in section 7.3 followed by sample extracts interspersed with mid-concentration calibration standards. Before any samples are analyzed the instrument must be calibrated by analyzing a six-point calibration or a 1.0ppm concentration standard (CV-calibration verification standard) for Aroclor 1660, Aroclor 1242, Aroclor 1248 and Aroclor 1254. If a CV is run, the calculated concentration must not exceed a difference of ± 15%. For clients or projects requiring DoD QSM 4.1, the response for any analyte must not vary from the expected response by more than ± 20%, or a new calibration curve must be prepared for that analyte. If the CCV fails the above criteria, reanalyze all samples since the last successful calibration verification. If reanalysis cannot be performed, data must be qualified and explained in the narrative. Additionally, apply a Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification. Each sample analysis must be bracketed with an acceptable initial

calibration or an opening CV and an ending CV for each 12-hour shift. The closing CV for Aroclor 1660 is a 0.25ppm concentration standard. All other Aroclors at the closing of the run remain at 1.0ppm concentration. If a second window of samples is run immediately after the closing CVs, the concentration of Aroclor 1660 at the completion of this window would be 1.0ppm. The calibration standard must also be injected at intervals of not less than once every ten samples and at the end of the analysis sequence. If the CV fails, the instrument is checked for any obvious problems and maintenance is performed if deemed necessary. Another CV is analyzed or the instrument is recalibrated and then samples are injected. All samples that were injected after the standard exceeding the criterion must be reinjected to avoid errors in quantitation, if the initial analysis indicated the presence of the specific target analyte that exceeded the criterion.

7.5.3.1 However, if the standard analyzed after a group of samples exhibits a response for an analyte that is above the acceptance limit, i.e. >15%, and the analyte was not detected in the specific samples analyzed during the analytical shift, then the extracts for those samples do not need to be reanalyzed, as the CV standard has demonstrated that the analyte would have been detected were it present. In contrast, if an analyte above the QC limits was detected in a sample extract, then re-injection is necessary to ensure accurate quantitation. If an analyte was not detected in the sample and the standard response is more than 15% below the initial calibration response, then re-injection is necessary to ensure that the detector response has not deteriorated to the point that the analyte would not have been detected even though it was present.

7.5.4 The center of the retention time window for each analyte and surrogate is established by using the absolute retention time for each analyte and surrogate from the daily opening calibration verification or initial calibration.

7.5.5 The identification of PCBs is based on agreement between the retention times of peaks in the sample chromatogram with the retention time windows established through the analysis of standards of the target analytes. An analyte is tentatively identified when a peak from a sample falls within the daily retention time window. Each tentative identification must be confirmed using a second GC column of dissimilar stationary phase or using another technique such as GC/MS. If the retention times of the peaks on both columns fall within the retention time windows on the respective columns, then the target analyte identification has been confirmed.

7.5.5.1 An additional criterion is applied for the identification and quantitation of PCBs. Identification is based on the characteristic fingerprint

retention time and shape of the major peaks. Major peaks are defined as those peaks in the Aroclor standard that are at least 25% of the height of the largest Aroclor peak. The sample chromatogram is compared to the individual Aroclor standard chromatograms. Once the Aroclor pattern has been identified, a concentration is then calculated in Target.

7.5.5.2 Three to five Aroclor concentrations are calculated using the peak heights of the three to five characteristic peaks of the Aroclor. These three to five concentrations are then averaged to determine the concentration of that Aroclor.

7.5.6 When samples are analyzed from a source known to contain specific Aroclors, the results from a single-column analysis may be confirmed on the basis of a clearly recognizable Aroclor pattern.

7.5.7 If the response for an analyte exceeds the calibration range of the system, the sample must be diluted and reanalyzed.

7.5.8 If peak detection and identification are prevented due to interferences, the hexane extract may need to undergo a cleanup. The extract may be subjected to a sulfur cleanup (method 3660) and/or a sulfuric acid cleanup (method 3665).

Note: Samples routinely receive a sulfuric acid clean up. However, for samples from a known site with a clean matrix, a sulfuric acid clean up may not be performed. Whenever a sample receives a cleanup, the associated QC must also be subjected to the same cleanup(s) and reanalyzed.

7.5.9 When a GC system is determined to be out of control because either a CV cannot pass or a six point calibration does not meet the correlation coefficient criteria, instrument maintenance is likely necessary. Routine instrument maintenance may involve changing the septum, replacing the liner, clipping the pre-column, or replacing the column. This information is recorded in the instrument run log (Figure 1). When an instrument requires more severe maintenance like replacing the ECD or an electronic board, this information is written in the instrument maintenance logbook.

7.6 Calculations

7.6.1 The concentration of an analyte is calculated by using the calibrated curve that is prepared in Target. When an analyte is identified, Target displays a concentration after the file is processed through the appropriate calibration method. Aroclor quantitation is accomplished by the method described in

section 7.5.4.1.1. However, if a sample contains more than one Aroclor, a peak common to both analytes must not be used to quantitate either compound.

7.6.2 The concentrations from the reports are then incorporated with the extraction data to arrive at a final concentration.

Water: Concentration (ug/L) = (C) (Vt)/ (Vs)

Soil/Sediment: Concentration (mg/kg) = (C) (Vt)/ (Ws) (D)

where, C = concentration calculated by Target in ug/ml
Vt = Volume of total extract including any instrument dilutions
Vs = Volume of sample extracted
Ws = Weight of sample extracted
D = Decimal total solids

7.7 Data Review

7.7.1 Initial Data Review

The initial data review is accomplished by the analyst who ran the samples. This review is of sufficient quality and detail to provide a list of samples that need to be reanalyzed or diluted and reanalyzed. The initial data review is performed on the detailed quantitation reports of the analyzed samples. This data review examines criteria that directly impact whether or not the sample needs to be reanalyzed and/or extracted. These criteria include:

- QC criteria for method blank, LCS, MS/MSD, and calibration – refer to section 8.0.
- Surrogate recovery
- Chromatography: cleanups, manual integration.
- Target compound detection: quantitation, confirmation, false positives.

The requirement of the GC laboratory is that this initial data review be completed no later than the end of the next workday. After the analyst has completed his or her initial data review, the information is then ready to be processed for reporting. Refer to section 7.8.

7.7.2 Surrogate recovery

All recoveries must meet the most recently laboratory established acceptance limits, which are listed on the GC Laboratory Surrogate Acceptance Limit sheet.

The sample is evaluated for recoveries of the two surrogates. If the recovery of one surrogate is within the acceptance limit, and the second is out, the data is narrated. If the surrogate recoveries are high for both and the sample contains less than the PQL for all target analytes, the data is narrated. If the surrogate recoveries are low and may be attributable to matrix interference or a matrix effect, the data is narrated. If the surrogate recoveries are low and the sample concentration is less than the PQL for all target analytes and there is no apparent matrix effect, reextract the sample.

For method blanks, if the recoveries of both surrogates are low or high, and the blank does not contain any target analytes above the PQL, and the recoveries of both surrogates in the sample(s) are acceptable, the data is narrated. If the recoveries in the blank are low and it does not contain any target analytes above the PQL, and the recoveries in the samples are acceptable but the sample contains one or more target analytes above the PQL, the sample may be reextracted.

For laboratory control samples (LCS), if the only discrepancy in the extraction batch is with the LCS, and the analyte spike recoveries are acceptable, the data is narrated. If the recoveries of both the surrogates and the analyte spikes are low, the samples may need to be reextracted.

For DoD QSM 4.1, use QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. When the surrogate recoveries fall outside of the acceptance criteria, apply Q-flag to all associated analytes.

7.7.3 Chromatography

The chromatography should be examined for the presence of any non-target peaks, which can be used as an indication of whether or not matrix interference might be influencing surrogate recoveries. If the chromatogram indicates interferences, then a cleanup may be needed. See section 7.5.7.

Manual integrations are to be performed when chromatographic conditions preclude the computer algorithm from correctly integrating the peak of concern. In no instance shall a manual integration be performed solely to bring a peak within criteria.

Each peak of concern is examined by the primary analyst to ensure that the peak was integrated properly by the computer algorithm. Should a manual integration be necessary (for instance, due to a split peak, peak tailing, or incomplete resolution of isomeric pairs), an “m” qualifier will automatically be printed on the quantitation report summary. The analyst will date and initial the “m” on the quanitation report summary and assign a code that indicates

the reason for the manual integration. Refer to Katahdin SOP QA-812 "Manual Integration on GC/MS, GC, HPLC and IC Datasystems" for more information.

7.7.4 Target Compound Detection

GC analysis relies heavily on the experience of the analyst. Sample chromatograms must be evaluated focusing on scientific judgment, knowledge of the column behavior and matrix effects. The chromatogram from channel A is evaluated with that from channel B. If a target analyte is present on both channels and the concentration is within the calibration range, and the quantitation from both chromatograms agrees within ±40%, the analyte is considered present in the sample. In cases where the RPD is greater than 40% and the analyte is reported, the analyte must be J-flagged and narrated. The higher of the two concentrations is reported unless matrix interference is causing erroneously high results. In this case report the lower result and narrate. In some cases a non-confirming analyte may be reported. In these cases the analyte must be Q-flagged and narrated...

In order to avoid reporting false positives, identified peaks on a chromatogram may need to be undetected electronically in Target. The possible scenarios are: If an analyte is present on one column but its concentration is below the PQL, if an analyte is present on one column but does not confirm on the other channel, if an analyte is present on both columns but the concentrations differ by more than 40%, or if an analyte is present but its retention time is ±0.04 minutes or more than the retention time of the analyte in the preceding CV. The GC Analyst must rely on technical experience in reviewing chromatograms in determining if a hit is an actual analyte or a false positive.

If reporting data that has an RPD that is >40%, the data must be flagged with a "J" indicating that the result is an estimated value. Sometimes interference on one column (i.e. sulfur) will prevent a target analyte from detection and it is present on the conformational column. In this scenario, the result would be reported from one column and need to be "Q" flagged to indicate that it was not confirmed on a second column.

7.7.5 Reporting

After the chromatograms have been reviewed and any target analytes have been quantitated using Target, the necessary files are brought into KIMS. Depending on the QC level requested by the client, a Report of Analysis (ROA) and additional reports, such as LCS forms and chronology forms, are generated. The package is assembled to include the necessary forms and raw data. The data package is reviewed by the primary analyst and then forwarded

to the secondary reviewer. The secondary reviewer validates the data and checks the package for any errors. When completed, the package is sent to the Department Manager for final review. A completed review checklist is provided with each package. The final data package from the Organics department is then processed by the Data Management department.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

Refer to Table 1 and to details in this section for a summary of QC requirements, acceptance criteria, and corrective actions. These criteria are intended to be guidelines for analysts. The criteria does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in this section or in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in this section and in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations, client and project specific Data Quality Objectives and on review of chromatograms. The supervisor, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

8.1 For each analytical batch (up to 20 samples), a method blank, laboratory control sample (LCS), matrix spike and matrix spike duplicate are analyzed. They are carried through all stages of the sample preparation and analysis steps.

8.2 Spike concentrations: The LCS and the MS/MSD are spiked at the same concentration with Aroclor 1660. The spike concentrations are:

Compound	WATER ug/L	SOILS mg/kg
Aroclor 1660	5.0	0.17

The surrogate spike concentrations in the final extract are:

Compound	WATER ug/ml	SOILS ug/ml
Tetrachloro-m-xylene(TCX)	0.10	0.10
DCB	0.10	0.10

8.3 LCS and MS/MSD acceptance criteria and Corrective Action: All QC samples are calculated for percent recovery of the spiked analyte. The recoveries are compared to laboratory established acceptance limits. The LCS acceptance limits for PCBs are established for both water and soil matrices. The MS/MSD acceptance limits for PCBs use the respective matrix LCS acceptance limits. Separate limits for MS/MSD pairs are not calculated because of the varying matrices involved. In addition many of the MS/MSD data points cannot be used (i.e. recoveries not calculable due to a matrix effect).

If any spike compound in the laboratory control sample falls outside of the established recovery acceptance limit window, the QC sample is considered to be out of control and any sample that is associated should be evaluated with other QC elements to determine the corrective action. If the recovery is high and the associated samples do not contain the specific compound(s), the data can possibly be accepted with narration. In other cases, the associated samples must be extracted.

If a spike compound is outside of the acceptance limits in the matrix spike sample but is acceptable in the LCS, the data is considered acceptable. The cause of the failure is possibly attributable to matrix interference. However, if the compound fails in both the LCS and the MS/MSD, the result for that analyte is suspect and may not be reported for regulatory compliance purposes.

For DoD QSM 4.1, use QC acceptance criteria specified by DoD, if available. Otherwise use in-house control limits. In-house control limits must not be greater than $\pm$ 3 times the standard deviation of the mean LCS recovery. If the LCS fails the acceptance criteria, correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. If reanalysis cannot be performed, data must be qualified and explained in the narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.

For MS, when applying DoD QSM 4.1, apply J-flag to specific analyte(s) also in parent sample, if acceptance criteria not met. RPD must be </= 30% between MS and MSD.

8.4 Surrogate acceptance criteria and Corrective Action: Surrogate recoveries are calculated on all samples, blanks and spikes. The recoveries are compared to laboratory established acceptance limits.

When a sample has a surrogate that falls outside of the laboratory established acceptance limit window, the problem should be investigated. If the recovery looks like it

is affected by the sample matrix, the sample may be reinjected to confirm matrix interference. When a sample has no detectable surrogate recovery, the sample should be reextracted.

For DoD QSM 4.1, use QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits. When the surrogate recoveries fall outside of the acceptance criteria, apply Q-flag to all associated analytes.

8.5 Non-conformance Report: Whenever data is not acceptable because of a failing LCS or surrogate recovery, a non-conformance report (NCR) must be initiated as soon as possible to document resolution.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs are determined prior to sample analysis per type of instrument and filed with the Organic Department Manager and with the QAO. Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 8082 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition. Final Update IV, dated February, 2007, Method 8082A.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Current Version.

Katahdin Analytical Services, Inc., SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

Katahdin Analytical Services, Inc., SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF FIGURES

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 1

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
6pt calibration of Aroclor 1660, 1242, 1248, 1254 and mid-point cal of other Aroclors	Initial cal prior to sample analysis	Average Model – at least 5 points, % RSD </= 20% Linear Model – at least 5 points, correlation coefficient (r) $\geq$ 0.990 Quadratic Model – at least 6 pt calibration, coefficient of determination (r^2) $\geq$ 0.990	(1) Repeat Initial calibration (2) If single pt cal Aroclor is identified in analysis of sample,5 or 6-pt calibration (depending on calibration model) of identified compound with reanalysis of sample.
Independent Calibration Verification	Immediately following calibration	± 20 % D	(1) Reanalyze standard (2) Reprep standard (3) Reprep standard from fresh stock.
CCV	After every 10 samples; If calibration curve previously analyzed, analyze daily before samples.	± 15 % D	(1) Evaluate the samples: If the %D >+15% and sample results are <PQL, narrate. (2) If %D >$\pm$15% only on one channel, narrate. If %D >$\pm$15% for closing CV, and is likely a result of matrix interference, narrate. (3) Otherwise, reanalyze all samples back to last acceptable CV.
Method blank	One per prep batch	No analyte detected >PQL	(1) Investigate source of contamination (2) Evaluate the samples and associated QC: i.e. If the blank results are above the PQL, report sample results which are <PQL or > 10X the blank concentration. (3) Otherwise, reprep a blank and the remaining samples.
LCS	One per prep batch of twenty or fewer samples	Laboratory statistically derived limits.	(1) Evaluate the samples and associated QC: i.e. If an MS/MSD was performed and acceptable, narrate. (2) If an LCS/LCSD was performed and only one of the set was unacceptable, narrate. (3) If the surrogate recoveries in the LCS are also low but are acceptable in the blank and samples, narrate. (4) If the LCS recovery is high but the sample results are <PQL, narrate. (5) Otherwise, reprep a blank, QC and the remaining samples.
Matrix Spike\ Matrix Spike Duplicate	One for every set of 20 samples	Same as for LCS	(1) Evaluate the samples and associated QC: i.e. If the LCS results are acceptable, narrate. (2) If both the LCS and MS/MSD are unacceptable reprep samples and QC.

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 1 (cont.)

QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Sample Duplicate	One sample duplicate per ten samples if requested	RPD $\leq$20	(1) If lab QC in criteria and matrix interference suspected, flag data or narrate (2) Otherwise, reanalyze
Demonstration of analyst proficiency – 4 replicates	Once per analyst initially and annually thereafter	P&A meet method criteria	(1) Repeat P&A study
MDL study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

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TABLE 2

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	Not Applicable (NA).	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from a 72-hour study.	NA.	NA.	
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	6 point calibration of Aroclors 1016, 1242, 1248, 1254 and 1260 - One of the options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression: r ≥ 0.995; Option 3: non-linear regression: coefficient of determination (COD) r2 ≥ 0.99 (6 points shall be used for second order). Mid point calibration of Aroclors 1221 and 1232; if targets are detected, 6-point calibration is performed.	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin. Quantitation for multicomponent analytes such as chlordane, toxaphene, and Aroclors must be performed using a 5-point calibration. Results may not be quantitated using a single point.
Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	NA.	

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TABLE 2 (cont)

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification (ICV)	Immediately following ICAL.	All project analytes within established retention time windows. All project analytes within ± 20% of expected value from the ICAL.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All project analytes within established retention time windows. All project analytes within ± 20% of expected value from the ICAL.	Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported, including surrogates	One per preparatory batch.	The laboratory shall use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements. In-house CLs may not be greater than ± 3 times the standard deviation of the mean LCS recovery. A number of analytes may fall outside the CL but within marginal exceedance limit depending on the total number of analytes in the LCS.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available. Refer to Table G-1 for number of marginal exceedences allowed. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

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TABLE 2 (cont.)

DOD QSM REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix if sufficient sample is available.	For matrix evaluation, use laboratory control limits (CLs) or use DoD-generated LCS-CLs, if available depending on project requirements.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix Spike duplicate (MSD)	One per preparatory batch per matrix if sufficient sample is available.	MSD: For matrix evaluation, use laboratory LCS CLs or use DoD-generated LCS CLs, if available depending on project requirements. MS/MSD: RPD ≤ 30%.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Surrogate spike	All field and QC samples.	The laboratory shall use laboratory CLs or use DoD-generated Surrogate CLs, if available depending on project requirements.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary. Contact Client if samples cannot be reprepped within hold time.	Apply Q-flag to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed (with the exception of Method 8015).	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD ≤ 40%.	NA.	Apply J-flag if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method reporting requirements; otherwise, report the result from the primary column.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-329-13	METHOD 8082, current revision
Procedures	7.4.5 If the calculated retention time window results in a value of 0.03 minutes or less, the laboratory will apply nominal windows. This is done in order to avoid any false negative hits because of the window being to narrow. The windows are: ± 0.07 for all target analytes. By utilizing these windows, a false positive hit may be initially indicated, but an experienced analyst could determine a false positive from scrutinizing the chromatograms. Please note that the use of nominal retention time windows may not be allowable for certain states, federal programs, or clients. South Carolina does not allow the use of nominal limits for compliance work originating in their state. In these cases, a window of ± 0.03 minutes must be used if the established retention time window is less than 0.03 minutes.	9.3 refers to method 8000B section 7.6.3: If the standard deviation of the retention times for a target compound is 0.000 (i.e., no difference between the absolute retention times), then the laboratory may either collect data from additional injections of standards or use a default standard deviation of 0.01 minutes. (Recording retention times to three decimal places rather than only two should minimize the instances in which the standard deviation is calculated as 0.000).
Apparatus/Materials		
Reagents		
Sample Preservation and handling		
QC – Spikes		
QC – LCS		
QC – Accuracy/ Precision		
QC - MDL	PQL Practical Quantitation Level – three to ten times the MDL.	EQL Estimated Quantitation Level – five to ten times the MDL

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 1

EXAMPLE OF INSTRUMENT RUN LOG

Katahdin Analytical Services, Inc.

GC Laboratory Instrument Runlog
Instrument: GC07
Amount Injected: 2 uL
Column Numbers: 390/391

Method: SW846 8082 EPA 608
(circle)

Standard	Standard ID
AR1660 1.0	P6813
AR1242 1.0	P6795
AR1248 1.0	P6796
AR1254 1.0	P6792
AR1660 0.25	P6812

Date	Init.	Result File	Sample ID	Y/N	Analytical Workgroup	Method	Comments
			changed liner, clipped column, replaced septa				
2/16/15	CB	7487138	PHME	N		PCB057	instrument check
		139	AR1660 1.0				
		140	AR1242 1.0				
2/16/15	CB	7487141	PHME	N		PCB057	
		142	AR1660 1.0	Y	W4120137		TTCA, TCA, TCB, TTCA
		143	AR1242 1.0	Y			
		144	AR1248 1.0	Y			
		145	AR1254 1.0	Y			
		146	W4120093-1 3500				CC.2 3500uL/500uL
		147	-2				ES.10 sample+500uL IS@A
		148	S40944-1				ES.25 500uL+500uL
		149	-2				
		150	AR1660 0.25	Y			TTCA, TCA
		151	AR1242 1.0				
		152	AR1248 1.0				
		153	AR1254 1.0	Y			
2/16/15	CB	7487154	PHME	N		PCB057	
		155	AR1660 1.0	Y	W4120196-1.2		TTCA
		156	AR1242 1.0	Y			
		157	AR1248 1.0	Y			
		158	AR1254 1.0	Y			
		159	W4120052-1 3500				
		160	-2				
		161	-3				
		162	S40942-1				TTCA
		163	-2				TTCA
		164	-3				TTCA
		165	-4				TTCA
		166	S40824-1				TTCA
		167	-2				TTCA

GC-001 – Revision 2 – 07/25/2012

QAGC276

0000017

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 2

DATA REVIEW CHECKLIST

PRIMARY REVIEW CHECKLIST

Verbal Due Date _____ (☐ Verbals turned in). Due Date_____

Client:		Primary	Secondary
Method:		Date:	Date:
SDG No:	Level:	Initials:	Initials:
KAS No:			Approved : ☐ Yes

DODQSM (4.1) ☐ DOD W/ LAB. LIMITS ☐ QUAPP ☐ LAB ☐
(*REPORT ND's to - PQL* ☐ *MDL* ☐ *LOD* ☐)

List all curves that ***are scanned*** (hard copy not included). _____

_____ _____ _____ _____

Narrate which QC limits were used for (Surr., LCS's MS/MSD's.) ☐
All needed forms are present . ☐
Correct Work Order Number or SDG name (all forms). ☐
Correct project name and spelling (all forms). (Truncated ☐). ☐
Correct file numbers (all forms). ☐
Analysis Date Correct. ☐
Extraction Method & Analysis Method Correct. ☐
Product list compared to ROAs (compounds & PQLs). ☐
Chromatogram reviewed for unlabeled peaks (check product list). ☐
Flagging of all ROAs correct (Florida ☐) (Florida ☐). ☐
All tunes included (level IV) . ☐
All log book pages included (Soil weights,TCLP & SPLP). ☐
Verify DOD QSM criteria and/or Project specific requirements. ☐
Narrate any method deviations. (Blanks, LCS's etc.) ☐
Sign & Date Manual integration **(Narrate as needed).** ☐

Sample I.D's Truncated (**NARRATED**). YES ☐ Please list KAS # below :

_____

QA-044 – Revision 3 – 10/24/2012

TITLE: ANALYSIS OF PCBs AS TOTAL AROCLORS BY GAS CHROMATOGRAPHY/ ELECTRON CAPTURE DETECTOR (GC/ECD): SW-846 METHOD 8082

FIGURE 3

PQLs FOR METHOD 8082

ANALYTE	Practical Quantitation Level (PQL) (ug/L)	Practical Quantitation Level (PQL) (ug/kg)
PCB-1016	0.50	17
PCB-1221	0.50	17
PCB-1232	0.50	17
PCB-1242	0.50	17
PCB-1248	0.50	17
PCB-1254	0.50	17
PCB-1260	0.50	17
PCB-1262	0.50	17
PCB-1268	0.50	17

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES AND GFAA

Prepared By: George Brewer Date: 3/98

Approved By:

Group Supervisor: George Brewer Date: 01/24/01

Operations Manager: C. Burton Date: 1/24/01

QA Officer: Deborah J. Nadeau Date: 1.24.01

General Manager: Duncan F. Hughes Date: 1/25/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3050B	Format changes, added pollution prevention, added MSD, added spiking instruction tables.	DH	1.24.01	1/24/01
02 3050B	Removed all references/procedures devoted to GFAA. Added use of digestates for ICP-MS analysis. Revised standard solution names & concs. in Tables 3 & 4 to reflect current practice.	DH	8.29.02	8.29.02
03 3050B	New Title to include ILMO5.3. Use of digestion block and polyethylene digestion tubes added to sections 4.0, 7.0 and Table 1. PBS changed from 1.0g water to 1.0g boiling chips. H_2O_2 addition from 3.0ml then 1.0mls to 2.0ml, 2.0ml then 2.0ml figures and Tables updated to reflect current practices.	LAD	03/08	03/08
04	Updated Tables 3 and 4 with current spike concentrations and volumes added. Updated logbook page. Added CA-108 reference for subsampling information.	LAD	08/09	08/09
05	Updated Tables 3 and 4 to reflect current spiking procedures.	LAD	09/10	09/10

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-605-05**, titled **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS AND ILM05.3.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-605-05**, titled **ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS AND ILM05.3.**

Recipient: _____ Date: _____

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the Katahdin Analytical Services, Inc. procedure utilized to dissolve solid matrices and solubilize metals from solid samples prior to analysis for metals by ICP-AES and ICP-MS. This SOP applies to samples prepared by EPA Method 3050, with method modifications as summarized in Table 2.

This procedure applies to all solid sample (e.g. sediments, sludges, soils, and ashes) preparations for ICP-AES and ICP-MS analyses. This method is not a total digestion technique for most samples. It is a very strong acid digestion that will dissolve almost all elements that could become "environmentally available". By design, elements bound in silicate structures are not normally dissolved by this procedure as they are not usually mobile in the environment.

1.1 Definitions

ICP-AES – Inductively Coupled Plasma Atomic Emission Spectroscopy.

ICP-MS – Inductively Coupled Plasma Mass Spectrometry.

LCSS – Laboratory Control Sample for Solids – A standard or solid reference material that has been brought through the sample preparation process.

Matrix Spike – An aliquot of a sample to which a known amount of analyte has been added before digestion.

PBS – Preparation Blank for Solids – An aliquot of reagent water that has been brought through the sample preparation process.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of solid samples by USEPA Method 3050 for metals analysis. Each analyst must demonstrate the ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Training".

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of solid samples by USEPA Method 3050 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the procedure or irregularities with the samples should also be recorded in the lab notebook and reported to the responsible Department Manager or designated qualified data reviewer.

It is the responsibility of the Department Manager to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, aprons, dust masks, and shoe protectors, is available in the Metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully, while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever beakers are being heated on a hot plate. Use caution when handling hot beakers.

Personnel are required to read the Katahdin Hazrdous Waste Management Plan and Safety Manual before performing this procedure, and must be familiar with the general rules for laboratory safety, personal hygiene, housekeeping, and use of protective clothing and equipment.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Management Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the Metals prep lab for subsequent appropriate disposal in accordance with the Katahdin Hazardous Waste Mnagement Plan and Safety Manual.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

2.0 SUMMARY OF METHOD

A representative 1 to 2 g (wet weight) sample is digested with repeated additions of nitric acid and hydrogen peroxide. Hydrochloric acid is added to the initial digestate and the sample is refluxed. The digestate is then filtered and diluted to a final volume of 100 mL.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1 Digestion vessels. If digestion is performed using a hot plate, the appropriate digestion vessels are 100 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning" and CA-602, “Glassware Preparation and Sample Preservation for Trace Element Analyses”). If digestion is performed using a block digester, the appropriate digestion vessels are new 70 mL disposable graduated polyethylene digestion tubes with attached snap lids.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40 mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate or block digester, griddle, or other heating source - adjustable and capable of maintaining a temperature of $95^{o}C \pm 5^{o}C$. Heating sources must be numbered for easy identification.

4.6 Device for measuring hot plate temperature, consisting of a flask or digestion vessel in which the bulb of a thermometer is immersed in sand or water. The temperature of each hot plate used is measured and recordedeach day. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water, again allowing each rinse to drain completely. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, 1:1 HNO_3, and concentrated HCl.

4.13 Analytical balance capable of reading to 0.01 gram.

4.14 Spatulas, scoops, or spoons; plastic or stainless steel, rinsed with 5% HNO_3 and reagent water. Disposable tongue depressors may be used and do not requrire to be rinsed.

5.0 REAGENTS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Nitric acid, 1:1. Add a volume of concentrated HNO_3 to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 30% hydrogen peroxide (H_2O_2) - spectrometric grade.

5.7 Multielement spiking solutions (see Table 3 for a list of required spiking solutions).

5.8 Solid reference material – a soil containing all the elements of interest, with empirically established method-specific recoveries and acceptance limits for all analytes. Solid reference materials are purchased with documentation of analysis provided by the vendor. See Figure 4 for an example certificate of analysis for a solid reference material.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples should be collected in clean plastic or glass containers. Samples must be refrigerated ($4^oC \pm 2^oC$) upon receipt by the laboratory. The holding time for solid samples is 6 months from the date of sample collection.

7.0 PROCEDURE

The procedure described below is condensed for quick reference in Table 3.

SAMPLE PREPARATION

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet (see Figure 2 for an example). Hand label the digestate vessels

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers and watch glasses three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digeter do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 Weigh 1 to 2 g of well-mixed sample into a properly cleaned, labeled, and tared Griffin beaker or polyethylene digestion tube. Record (hand write) the weight of each sample on the printout of the digestion spreadsheet. Refer to Katahdin Analytical Services SOP CA-108, current revision “Basic Laboratory Technique” for more information on subsampling.

7.4 Weigh an appropriate amount of solid reference material to a clean, labeled, and tared Griffin beaker or polyethylene digestion tube to serve as a laboratory control sample.

7.5 Add spike solutions to matrix spike samples (refer to Tables 3 and 4 for spiking instructions).

7.6 Using repipetters, add 10 mL of 1:1 HNO_3, mix the slurry. Cover with a ribbed watch glass and place on heat source. Gently heat the sample to 95^{o}C ± 5 ^{o}C and reflux for 10 to 15 minutes without boiling. Remove the digestion vessel from the heat source and cool the sample.

7.7 Add 5 mL of concentrated HNO_3 to the sample, replace the watch glass, and reflux for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3, repeat this step (addition of 5 mL of concentrated HNO_3) until no brown fumes are given off by the sample, indicating complete reaction by HNO_3.

7.8 Continue heating the sample at 95^{o}C ± 5^{o}C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the digestion vessel from the heat source and cool the sample.

7.9 Add 2 mL of reagent water and 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.10 Add an additional 2 mL of 30% H_2O_2 to the sample, replace the watch glass, and heat gently on the heat source to start the peroxide reaction. Continue heating until effervescence subsides.

7.11 Add an additional 6 mL of 30% H_2O_2 in 1-mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged.

7.12 Continue heating the sample at 95^{o}C ± 5^{o}C without boiling until the digestate has evaporated to approximately 5 to 10 mL or until two hours have elapsed, whichever occurs first. Do not allow the sample to go to dryness. Remove the sample from the heat source and cool.

7.13 Add 10 mL of concentrated HCl to the digest from 7.12, replace the watch glass, and reflux at 95^{o}C ± 5^{o}C for 15 minutes. Remove the sample from the heat source and cool.

7.14 Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated polystyrene specimen container or graduated polyethylene sample container with attached snap lid. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse. Using the graduations on the specimen container or snap-lid container, dilute to 100 mL with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid

container has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for ICP-AES or ICP-MS analysis.

7.15 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) reagent lot numbers, spiking information, and heat source temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 2.

7.15 Reopen the electronic ACCESS spreadsheet for the digestion and transcribe the sample weights from the handwritten, bound copy into the electronic copy. The information in this electronic spreadsheet will later be imported into the ACCESS metals database and used to calculate sample concentrations on a weight basis.

7.16 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

CALCULATIONS

7.17 Analytical results for solid samples are reported on a dry weight basis. Total solids are determined by the Wet Chemistry Group, and are recorded in spreadsheets that are electronically imported into the Access metals database. Final dry weight concentrations are calculated by the Access database as follows:

$$\text{Concentration (mg/kg dry weight)} = (C \times V) / (W \times S)$$

where: C = Measured concentration (mg/L)
V = Digestate final volume (L)
W = Sample wet weight (kg)
S = % Solids/100

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 3050 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and

standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Depatment Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

8.1 At least one preparation blank for soils (PBS) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBS consists of a 1.0 g of boiling stones that is digested using the same reagents as those used to digest associated samples. Refer to the appropriate analytical SOP for PBS acceptance criteria and corrective actions.

8.2 At least one laboratory control sample for soils (LCSS) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSS consists of an aliquot of a solid reference material for which the concentrations of the analytes of interest have been empirically established (solid-matrix LCSS), or an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations (aqueous-matrix LCSS). The solid reference material should normally be used as the LCSS, unless a particular client or analytical program requires that spiked reagent water be used. The LCSS is digested using the same reagents as those used to digest associated samples. Directions for spiking the aqueous-matrix LCSS are contained in Table 3. The measured analyte recoveries for the LCSS are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSS recovery acceptance criteria and corrective actions.

8.3 Matrix spike samples are processed along with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is fortified with known amounts of all analytes of interest prior to digestion. Matrix spike recoveries are used to assess the biasing effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figure 2. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

NOTE: Clients may choose specific samples for matrix spike and duplicate analysis; otherwise, the choice is left to the person performing the digestion.

8.5 The quality control measures and frequencies described above are minimum requirements. Individual clients and analytical programs may impose additional QC requirements.

9.0 METHOD PERFORMANCE

Refer to the applicable instrumental analysis SOP for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

"Test Methods for the Evaluation of Solid Waste," United States Environmental Protection Agency, SW-846, Third Edition, Final Update III, 12/96, Method 3050B.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

LIST OF TABLES AND FIGURES

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 1

QC REQUIREMENTS – METHOD 3050

Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3050	Preparation Blank for Solids (PBS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Laboratory Control Sample for Solids (LCSS)	One per prep batch of 20 or fewer samples.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Matrix Spike Duplicate Sample	One per prep batch.	Refer to analytical method.	Refer to analytical method.
	Demonstration of analyst proficiency	One-time demonstration by each analyst performing the method.	Must pass all applicable QC for method.	Repeat analysis until able to perform passing QC; document successful performance in personal training file.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 2

SUMMARY OF METHOD MODIFICATIONS – METHOD 3050

Topic	Katahdin SOP CA-605-02	Method 3050, current revision
Apparatus /Materials	1) Digestion performed in 100 mL Griffin beaker or 70 mL polyethylene tube. 2) Graduated disposable plastic cup or 120 mL polyethylene tube used to bring digestate to final volume.	1) Digestion performed in 250 mL Griffin beaker. 2) Volumetric flask used to bring digestate to final volume.
Procedure	1) Digestate volume reduced to 5 to 10 mL prior to filtering. 2) After filtration, the filters are rinsed three times with reagent water. 3) 30% H_2O_2 is added in two 2 mL aliquots and then six 1 mL aliquots.	1) Digestate volume reduced to 5 mL prior to filtering. 2) After filtration, the filters are rinsed twice with reagent water. 3) 30% H_2O_2 is added in one 3 mL aliquot and then seven 1 mL aliquots.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 3

PREPARATION OF MATRIX SPIKES AND SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Matrix Spike for ICP-AES	CLPP-SPK-1	Inorganic Ventures(IV)	0.10
	CLPP-SPK-INT1	Lab Prepared (see below)	1.00
	CLPP-SPK-INT2	Lab Prepared (see below)	1.00
	1000 mg/L Uranium Std.	IV or High Purity Standards	0.01

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L As,Pb,Sb,Se,Tl	High Purity Standards	1.0 each
	1000 mg/L Cd	High Purity Standards	2.5
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
CLPP-SPK-INT2	1000 mg/L Mo	IV or High Purity Standards	3.0
	1000 mg/L B,Li,Sn,Sr,Ti	IV or High Purity Standards	5.0 each
	10000 mg/L Si	High Purity Standards	5.0

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

TABLE 4

ELEMENT CONCENTRATIONS IN ICP-AES MATRIX SPIKES AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF SOLID SAMPLES BY METHOD 3050

	CONCENTRATION IN SOLUTION, mg/L					
Element	**Matrix Spike**	**CLPP-SPK-1**	**CLPP-SPK-4**	**CLPP-SPK-INT1**	**CLPP-SPK-INT2**	**1000 mg/L U**
Aluminum	2.000	2000				
Antimony	0.100		100	10		
Arsenic	0.100		4	10		
Barium	2.000	2000				
Beryllium	0.050	50				
Boron	0.500				50	
Cadmium	0.250		5	25		
Calcium	2.500			250		
Chromium	0.200	200				
Cobalt	0.500	500				
Copper	0.250	250				
Iron	1.000	1000				
Lead	0.100		2	10		
Lithium	0.500				50	
Magnesium	5.000			500		
Manganese	0.500	500				
Molybdenum	0.300				30	
Nickel	0.500	500				
Potassium	10.000			1000		
Selenium	0.100		5	10		
Silicon	5.000				500	
Silver	0.050	50				
Sodium	7.500			750		
Strontium	0.500				50	
Thallium	0.100		5	10		
Tin	0.500				50	
Titanium	0.500				50	
Uranium	0.100					1000
Vanadium	0.500	500				
Zinc	0.500	500				

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 1

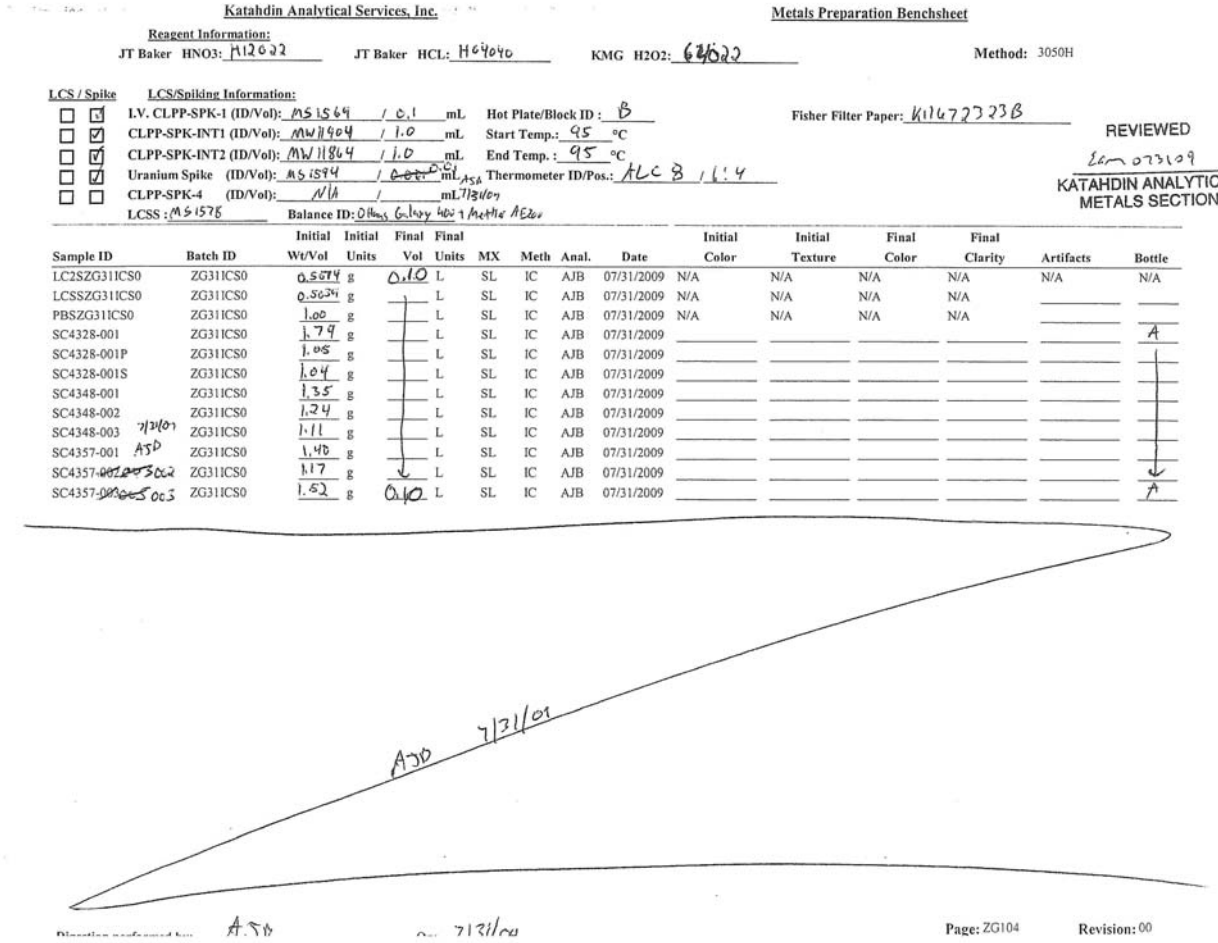
PROCEDURE CONDENSATION – METHOD 3050

1. Prepare and print out ACCESS spreadsheet.
2. If performing digestion on a hot plate, rinse 250 mL Griffin beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with laboratory reagent grade water. If perfoming digestion with block digester, polyethylene digestion tubes do not require precleaning.
3. Label digestion vessels (beakers or polyethylene sample tubes) with sample numbers.
4. Weigh 1 to 2 g of well-mixed sample into tared digestion vessels. Record sample weights.
5. Add spike solutions to matrix spike samples.
6. Add 10 mL 1:1 HNO_3 to samples and cover with watch glasses.
7. Reflux for 10 to 15 minutes at $95^{o} \pm 5^{o}$ C. without boiling. Cool samples.
8. Add 5 mL conc. HNO_3, cover beakers, and reflux for 30 minutes.
9. Repeat Step 8 as necessary until digestion is complete.
10. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
11. Cool sample and add 2 mL reagent water and 2 mL 30% H_2O_2. Heat gently until effervescence subsides.
12. Cool sample and add 2 mL 30% H_2O_2. Heat gently until effervescence subsides.
13. Cool samples and add 6 mL of 30% H_2O_2 in 1 mL aliquots. Heat gently until effervescence subsides.
14. Reduce sample volumes to 5 to 10 mL or heat for 2 hours, whichever occurs first.
15. Add 10 mL conc. HCl and reflux for 10 to 15 minutes at $95^{o} \pm 5^{o}$ C.
16. Cool sample and filter into graduated specimen container. Bring to volume with reagent water and transfer to labeled polyethylene bottle.
17. Enter sample weights into ACCESS spreadsheet.

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 2

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK



Katahdin Analytical Services, Inc. — Metals Preparation Benchsheet

Reagent Information: JT Baker HNO3: M12022 JT Baker HCL: H64040 KMG H2O2: 6/1/22 Method: 3050H

LCS / Spike — LCS/Spiking Information:

- LV. CLPP-SPK-1 (ID/Vol): MS1569 / 0.1 mL — Hot Plate/Block ID: B — Fisher Filter Paper: K1167223B
- CLPP-SPK-INT1 (ID/Vol): MW11904 / 1.0 mL — Start Temp.: 95 °C
- CLPP-SPK-INT2 (ID/Vol): MW11864 / 1.0 mL — End Temp.: 95 °C
- Uranium Spike (ID/Vol): MS1594 / ~~0.02~~ 0.01 mL ASA — Thermometer ID/Pos.: ALC 8 / 1.4
- CLPP-SPK-4 (ID/Vol): N/A / mL 7/31/09
- LCSS: MS1578 — Balance ID: Ohaus Galaxy 400 / Mettler AE200

REVIEWED Em 073109 KATAHDIN ANALYTICAL METALS SECTION

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Texture	Final Color	Final Clarity	Artifacts	Bottle
LC2SZG311CS0	ZG311CS0	0.5074	g	0.10	L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A	N/A	N/A
LCSSZG311CS0	ZG311CS0	0.5034	g		L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A		
PBSZG311CS0	ZG311CS0	1.00	g		L	SL	IC	AJB	07/31/2009	N/A	N/A	N/A	N/A		
SC4328-001	ZG311CS0	1.79	g		L	SL	IC	AJB	07/31/2009						A
SC4328-001P	ZG311CS0	1.05	g		L	SL	IC	AJB	07/31/2009						
SC4328-001S	ZG311CS0	1.04	g		L	SL	IC	AJB	07/31/2009						
SC4348-001	ZG311CS0	1.35	g		L	SL	IC	AJB	07/31/2009						
SC4348-002	ZG311CS0	1.24	g		L	SL	IC	AJB	07/31/2009						
SC4348-003 7/31/09	ZG311CS0	1.11	g		L	SL	IC	AJB	07/31/2009						
SC4357-001 AJD	ZG311CS0	1.40	g		L	SL	IC	AJB	07/31/2009						
SC4357-~~002~~003	ZG311CS0	1.17	g	↓	L	SL	IC	AJB	07/31/2009						↓
SC4357-~~003~~005	ZG311CS0	1.52	g	0.10	L	SL	IC	AJB	07/31/2009						A

AJD 7/31/09

Digestion performed by: AJD Date: 7/31/09 Page: ZG104 Revision: 00

TITLE: ACID DIGESTION OF SOLID SAMPLES BY USEPA METHOD 3050 FOR METALS ANALYSIS BY ICP-AES, ICP-MS

FIGURE 3

EXAMPLE CERTIFICATE OF ANALYSIS FOR SOLID REFERENCE MATERIAL

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

MS1475

DataPacK™
Lot No. D051-540

Trace Metals in Soil
Catalog No. 540

Certification

Method 3050 HNO3, H2O2, HCl Parameter	Total Concentration [1] (mg/Kg)	Certified Value [2] (mg/Kg)	Performance Acceptance Limits™ [3] (mg/Kg)
aluminum	55600*	7870	4630 - 11100
antimony	160	70.5	D.L. - 149
arsenic	316	289	234 - 344
barium	869	211	174 - 247
beryllium	60.9	54.4	45.2 - 63.6
boron	129	91.3	58.8 - 124
cadmium	114	101	82.9 - 119
calcium	9750*	3680	2970 - 4390
chromium	249	224	180 - 268
cobalt	113	101	82.7 - 119
copper	94.9	88.0	73.3 - 103
iron	24400*	15700	6610 - 24900
lead	184	158	129 - 187
magnesium	3780*	2260	1760 - 2750
manganese	703	420	343 - 497
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	69.6	55.5 - 83.7
nickel	137	120	99.1 - 141
potassium	33000*	3000	2200 - 3800
selenium	146	130	101 - 159
silver	127	104	68.9 - 139
sodium	15600*	1080	692 - 1470
strontium	326	113	90.5 - 135
thallium	106	94.0	72.8 - 115
tin	175	149	104 - 194
titanium	3100*	284	116 - 453
vanadium	151	111	85.1 - 137
zinc	311	272	215 - 329

Method 3050 HNO3, H2O2 Parameter	Total Concentration [1] mg/Kg	Certified Value [2] mg/Kg	Performance Acceptance Limits™ [3] mg/Kg
aluminum	55600*	7380	4440 - 10300
antimony	160	75.2	D.L. - 198
arsenic	316	284	225 - 343
barium	869	217	177 - 257
beryllium	60.9	53.6	42.7 - 64.5
boron	129	89.5	58.9 - 120
cadmium	114	103	83.6 - 122
calcium	9750*	3540	2800 - 4270
chromium	249	224	172 - 275
cobalt	113	101	82.0 - 120
copper	94.9	85.5	70.4 - 100
iron	24400*	12500	5480 - 19500
lead	184	162	132 - 192
magnesium	3780*	2160	1650 - 2670
manganese	703	415	330 - 500
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	68.8	52.7 - 84.9
nickel	137	119	98.5 - 140
potassium	33000*	2840	2160 - 3520
selenium	146	135	104 - 166
silver	127	107	49.8 - 164
sodium	15600*	1010	709 - 1310
strontium	326	111	89.0 - 133
thallium	106	99.3	76.8 - 122
tin	175	148	70.6 - 225
titanium	3100*	283	104 - 463
vanadium	151	104	70.5 - 138
zinc	311	275	222 - 328

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Nicholas Tillotson

Review Date: 2/2/12

SOP Number: CA605

SOP Title: 3050 DIG

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 02/02/12

QAO Signature: Leslie Dimond **Date:** 020912

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Nicholas Tillotson

Review Date: 1/22/13

SOP Number: CA-605

SOP Title: Acid digestion of solid samples by USEPA method 3050 for metals analysis by ICP

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 02/26/13

QAO Signature: Leslie Dimond **Date:** 02/26/13

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Prepared By: George Brewer Date: 11/97

Approved By:

Group Supervisor: George Brewer Date: 01/19/01

Operations Manager: John C. Buxton Date: 1/22/01

QA Officer: Deborah J. Nadeau Date: 1·22·01

General Manager: Dennis F. Keefe Date: 1/22/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01 3010A	Format changes, added pollution prevention, block digester; revised database references; revised and added tables.	DN	1·22·01	1/22/01
02 3010A	Added wording allowing use of digestates for ICP-MS analysis. Added use of block digester as primary heating source & adjusted volumes. Revised standard solution names & concs. in Figures 3 & 4.	DN	8·29·02	8·29·02
03	Added Uranium to spiking solutions for LCS & MS/D. Removed the Internal Custody Record for Metals Digestates figure and reference.	LAD	04/06	04/06
04	Minor changes to Section 7 to reflect current practices. Updated Figure 1 - Sample Prep Logbook. Updated Figure 2 and 3 - Spike amounts.	LAD	05/09	05/09
05	Added references. Updated Figure 2 and 3 with correct spike information. Added CA-108 reference for subsampling information.	LAD	04/10	04/10

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ____ of document **SOP CA-604-05**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ____ of document **SOP CA-604-05**, titled **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**.

Recipient: _____ Date: _____

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure utilized by Katahdin Analytical Services, Inc. personnel to solubilize metals in aqueous samples, wastes that contain suspended solids, and mobility-procedure extracts prior to analysis by inductively coupled plasma atomic emission spectroscopy (ICP) and inductively coupled plasma mass spectrometry (ICP-MS). This SOP applies to samples prepared by EPA Method 3010, with the method modifications mentioned in Table 2.

1.1 Definitions - none.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the acid digestion of aqueous samples by EPA Method 3010. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in the acid digestion of aqueous samples using EPA Method 3010 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their work in the appropriate lab notebook. Any deviations from the method or irregularities with the samples should also be recorded in the lab notebook and reported to the Supervisor or designated qualified data reviewer responsible for these data.

It is the responsibility of the Supervisor to ensure that technical personnel perform acid digestions in accordance with this SOP and to confirm that their work is properly documented through periodic review of the associated logbooks.

1.3 Safety

The acids used in this procedure are highly corrosive and reactive, and spiking standards contain toxic metals. The toxicity and reactivity of client samples are usually unknown, so samples should always be assumed to present a contact hazard. To reduce or eliminate exposure to potentially harmful chemicals, lab coats, gloves, and safety glasses or goggles must be worn whenever handling samples or reagents. Additional safety apparel, including face shields, rubber aprons, dust masks, and rubber shoe protectors, is available in the metals prep lab and should be worn whenever circumstances warrant.

Acids should be added to samples slowly and carefully while watching for reactions. This should be done under a hood, in case harmful fumes are evolved.

Hood sashes should be lowered as far as possible whenever beakers are being heated in the hood. Use caution when handling hot beakers.

Each qualified analyst or technician must be familiar with Katahdin Analytical Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their supervisor, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Excess spiking solutions must be emptied into the corrosive waste carboy located in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Sample digestates should be stored for a minimum of 60 days after digestion to allow for analysis, and reanalysis if necessary. Digestates older than 60 days may be emptied into the corrosive waste carboy in the metals prep lab for subsequent appropriate disposal in accordance with the Chemical Hygiene Plan and Safety Manual.

Any other wastes generated during the preparation of samples must be disposed of in accordance with the Katahdin Chemical Hygiene Plan and Safety Manual and SOP SD-903, “Sample Disposal,” current revision.

2.0 SUMMARY OF METHOD

The aqueous sample is refluxed with nitric acid in a covered digestion vessel. Additional nitric acid is added until the color of the digestate has stabilized. After the digestate has been evaporated to a low volume, it is refluxed with hydrochloric acid and diluted to the appropriate final volume with reagent water.

Samples may be concentrated (i.e. final digestate volume less than initial sample volume) during digestion if lower detection limits are required. Volumes of reagents and spiking standards must be added in proportion to the final volume of the digestate. Because concentration of samples during digestion increases the concentrations of dissolved solids

and may exacerbate analytical interferences, concentration factors greater than 5 are not recommended.

3.0 INTERFERENCES

Interferences are discussed in the applicable analytical SOPs.

4.0 APPARATUS AND MATERIALS

4.1. 250 mL and 400 mL pre-cleaned Griffin beakers (cleaned according to the current revision of SOP CA-100, "Labware Cleaning") for digestion using a hot plate. If digestion will be performed using a block digester, 70ml graduated, polyethylene block digester tubes (with attached snap caps) will be used instead of glass beakers.

4.2 Ribbed watch glasses. If digestion is performed using a hot plate, 75 mm diameter and 100 mm diameter glass watch glasses (pre-cleaned as above) are used. If digestion is performed using a block digester, 40mm diameter disposable polyethylene watch glasses are used.

4.3 Adjustable volume automatic pipets covering the range from 10 uL to 1000 uL and disposable pipet tips; calibrated Finn pipets or Eppendorf pipets are acceptable.

4.4 Disposable graduated polystyrene specimen containers with pouring lips, 200 mL capacity.

4.5 Hot plate, block digester, or other heating source - adjustable and capable of maintaining a temperature of 90-95^{o}C. Hot plates must be numbered for easy identification.

4.6 Device for measuring hot plate temperature. This may consist of a heat-resistant 100ml beaker containing reagent water in which a thermometer is immersed. When using a block digester, a digestion tube containing reagent water in which a thermometer is immersed may be used. The temperature of one hot plate is measured each day, on a rotating basis. The hot plate identification number and the measured temperature are recorded on the sample preparation logbook sheet.

4.7 Plastic funnels, pre-cleaned as in Section 4.1.

4.8 Filter funnel holders, capable of suspending plastic funnels above disposable specimen containers.

4.9 Polyethylene wash bottles for dispensing reagent water and 5% HNO_3.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

4.10 Filter paper, Whatman No. 41 or equivalent. Filters are acid-washed immediately prior to use as follows. Place a pre-cleaned funnel in the funnel holder and put a disposable plastic specimen container under the funnel to collect the rinsates. Place a folded filter in the funnel and rinse three times with approximate 10 mL volumes of 5% HNO_3, making sure the entire surface of the filter is wetted each time and allowing each rinse to drain completely before continuing. Then rinse three times with approximate 25 mL volumes of reagent water. Discard the rinsates into the appropriate waste container. The acid-washed filter is now ready for use.

4.11 Polyethylene sample containers with screw caps or graduated polyethylene sample containers with attached snap lids, 125 mL capacity. These are not necessary when using the block digester since the final digestates are stored in the digestion tubes.

4.12 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid and 1:1 HCl.

5.0 REAGENTS

5.1 Concentrated nitric acid, HNO_3 – trace metals grade.

5.2 Concentrated hydrochloric acid, HCl – trace metals grade.

5.3 Reagent water - water that meets the performance specifications of ASTM Type II water (ASTM D1193).

5.4 Hydrochloric acid, 1:1. Add a volume of concentrated hydrochloric acid to an equivalent volume of reagent water and swirl gently to mix.

5.5 Nitric acid, 5% v/v. Add 25 mL concentrated HNO_3 to 475 mL reagent water in a 500 mL wash bottle. Cap, point the dispensing tip into a sink, and shake gently to mix.

5.6 Multi-element spiking solutions (as listed in Figure 3).

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for dissolved metals should be filtered through a 0.45 um membrane filter and preserved as soon as possible after collection. Samples to be analyzed for total metals should be preserved, unfiltered, as soon as possible after collection. Aqueous samples are preserved by acidification with nitric acid to a pH of <2.

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Please refer to Katahdin Analytical Services SOP CA-108, "Basic Laboratory Technique", current revision, for information on subsampling.

7.0 PROCEDURES

7.1 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer spreadsheet. Print out a copy of the spreadsheet. With a permamament marker, make sample labels and attach to the polyethylene sample containers that will contain the digestates.

7.2 If using glass beakers as the digestion vessels, submerge previously cleaned beakers three times into a 10% nitric acid bath, then rinse three times with reagent water. The polyethylene digestion tubes used in conjunction with the block digester do not require acid rinsing or precleaning. Label the digestion vessels with sample numbers.

7.3 If digestion is performed using a block digester, the sample aliquot may be measured in the digestion vessel using the graduations on the digestion tubes. Measure 50 ml of well-mixed sample into a 70 ml block digestion tube. A larger sample aliquot may be used (up to 250 mL) if concentration of the sample during digestion is desired. Sample volumes larger than 50 mL may be digested in 250 mL beakers. Measure aliquot of well-mixed sample into a graduated specimen cup and transfer into a properly cleaned 250 mL beaker. Sample volumes of more than 50ml may not be digested using the 70ml block digester tubes. The volumes of reagents and spiking solutions used must be adjusted in proportion to the final digestate volume. The reagent and spiking solution volumes listed below are based on a final volume of 50 mL.

7.4 Add spike solutions to matrix spike samples and laboratory control samples (refer to Figure 3 for spiking instructions).

7.5 Use a repipetter to add 1.5 mL of concentrated HNO_3 (per 50 mL final volume) to the sample. Cover with a ribbed watch glass and place on heatsource. Heat cautiously, without boiling the sample, and evaporate to a low volume (10 - 15 mL).

NOTE: Do not allow any portion of the bottom of the digestion vessel to go dry during any part of the digestion. If a sample is allowed to go to dryness, low recoveries may result. Should this occur, discard the digestate and re-prepare the sample.

7.6 Cool the sample and add another 1.5 mL aliquot (per 50 mL final volume) of concentrated HNO_3. Cover and resume heating, increasing the temperature until a gentle reflux action occurs.

7.7 Continue heating, adding additional acid as necessary, until the digestate is light in color or does not change in appearance with continued refluxing.

7.8 Evaporate digestate to a low volume (10 - 15 mL).

7.9 Cool the sample and use a repipetter to add 5 mL (per 50 mL final volume) of 1:1 HCl. Cover the sample and resume heating, refluxing for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

7.10 Allow the sample to cool.

7.11 If the digestate contains visible particulate material, it must be filtered. Use a pre-cleaned funnel and acid-rinsed filter paper to filter the digestate into a clean graduated plastic specimen container or block digester digestion tube. Using a wash bottle, rinse the digestion vessel with reagent water and add the rinsates to the filter apparatus. After all of the liquid in the filter has drained into the specimen container or digestion tube, thoroughly rinse the filter three times with small (5-10 mL) volumes of reagent water, allowing the liquid to drain completely after each rinse.

If the digestion was performed using hot plates and the digestate does not contain particulate material, simply decant the digestate into a clean graduated specimen container (or graduated sample container with attached snap lid), rinse the beaker with reagent water, and add the rinsates to the container.

If the digestion was performed using a block digester and the digestate contains no visible particulate material, the digestate may be brought to final volume and stored in the digestion tube without decanting or rinsing.

7.12 Using the graduations on the specimen container, snap-lid container or digestion tube, dilute to the required final volume with reagent water. If a specimen container has been used, transfer the contents to the corresponding labeled polyethylene sample bottle, cap the bottle, and discard the empty specimen container. If a snap-lid container or digestion tube has been used, close and secure the snap-lid. Shake the container gently to mix. The digestate is now ready for analysis.

7.13 Review the ACCESS computer spreadsheet for accuracy. If any information is incorrect, make the necessary changes to the computer spreadsheet and print out a corrected copy. Do not discard the original copy of the spreadsheet. Record (hand write) the sample bottle ID, reagent lot numbers, spiking information, initial and final volumes, hot plate ID and hot plate temperature in the appropriate spaces on the spreadsheet. Record any method deviations, irregularities with the samples, or other pertinent observations at the bottom of the page, and sign and date the spreadsheet. Bind all copies of the spreadsheet in the sample preparation log. An example sample preparation logbook page (ACCESS spreadsheet) is included as Figure 1.

7.14 Place each batch of digestates in a box labeled with the QC Batch ID, and put the box of digestates in the metals digestates storage area.

7.15 A condensation of the procedure described above is included in this SOP as Table 3. A controlled copy of this table may be posted in the metals preparation laboratory for reference by the analyst.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

8.1 At least one preparation blank for waters (PBW) is processed concurrently with each digestion batch of 20 or fewer samples, and is used to assess contamination resulting from the digestion procedure. The PBW consists of an aliquot of reagent water that is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the PBW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the PBW must also be concentrated). Refer to the appropriate analytical SOP for PBW acceptance criteria and corrective actions.

8.2 At least one laboratory control sample for waters (LCSW) is processed concurrently with each digestion batch of 20 or fewer samples. The LCSW consists of an aliquot of reagent water that is spiked to contain all analytes of interest at known concentrations, and is digested using the same reagents as those used to digest associated samples. The initial and final volumes of the LCSW must be identical to those of the associated samples (i.e., if the associated samples were concentrated during digestion, the LCSW must also be concentrated). Directions for spiking the LCSW are contained in Figures 3 and 4. The measured analyte recoveries for the LCSW are used to assess digestion method performance. Refer to the appropriate analytical SOP for LCSW recovery acceptance criteria and corrective actions.

8.3 Matrix spiked samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. A matrix spike sample consists of an aliquot of a sample that is spiked with known amounts of all analytes of interest. Matrix spike recoveries are used to assess the effects of sample matrix on digestion and analysis performance. Directions for spiking matrix spike samples are contained in Figures 3 and 4. Refer to the appropriate analytical SOP for matrix spike recovery acceptance criteria and corrective actions.

8.4 Matrix spiked duplicate samples are processed concurrently with each digestion batch at a minimum frequency of one per digestion batch. Matrix spiked duplicate samples are used to assess the precision of the digestion and analysis methods. Refer to the appropriate analytical SOP for matrix spike duplicate precision acceptance criteria and corrective actions.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

NOTE: Clients may choose specific samples for matrix spike and matrix spike duplicate analysis; otherwise, the choice is left to the person performing the digestion. The sample volumes available may restrict the choice of samples used for matrix spike and duplicate digestion. Field blank samples should not be chosen for matrix spike and matrix spike duplicate analysis.

8.5 The quality control measures and frequencies described above are minimum requirements. They are summarized for reference in Table 1. Individual clients and analytical programs may impose additional QC requirements.

9.0 METHOD PERFORMANCE

Refer to the applicable analytical SOPs for method performance information.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 3010A.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Version 4.1, 04/22/09.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

LIST OF TABLES AND FIGURES

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 1

QC REQUIREMENTS

Analytical Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
3010	Preparation Blank for Waters (PBW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Laboratory Control Sample for Waters (LCSW)	One per prep batch of 20 or fewer samples	Refer to analytical method	Refer to analytical method
	Matrix Spike Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Matrix Spike Duplicate Sample	One per prep batch	Refer to analytical method	Refer to analytical method
	Demonstration of analyst proficiency; accuracy and precision	One time demonstration by each analyst performing the method	Must pass all applicable QC for method	Repeat analysis until able to perform passing QC; document successful performance in personal training file

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 2

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-604-05	EPA METHOD 3010, current revision
Apparatus/Materials	1) Disposable plastic specimen cup used to measure sample volume. 2) Digestion performed in 250 mL, 400 mL Griffin beaker, or 70ml digestion tube to facilitate evaporation. 3) Ribbed watch glass used throughout digestion to reduce contamination.	1) Graduated cylinder used to measure sample volume. 2) Digestion performed in 150 mL Griffin beaker. 3) Ribbed and non-ribbed watch glasses alternated in digestion.
Procedures	1) Digestate may be analyzed for antimony and silver. 2) Sample aliquots larger or smaller than 100 mL may be used. 3) Sample evaporated to 10 - 15 mL.	1) Digestate may not be analyzed for antimony and silver. 2) Requires sample aliquot of 100 mL. 3) Sample evaporated to 5 mL.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

TABLE 3

PROCEDURE CONDENSATION: EPA METHOD 3010

1. If performing digestion on a hot plate, rinse glass beakers and ribbed watch glasses 3 times in acid bath. Then rinse beakers and watch glasses 3 times with reagent water. If performing digestion with block digester, polyethylene digestion tubes do not require precleaning.
2. Label digestion vessels with sample numbers.
3. Mix sample well, measure 50 mL (or smaller or larger aliquot) into a polyethylene digestion tube. If using glass beakers, measure aliquot into graduated specimen container, and transfer to appropriate digestion vessel.
4. Add spike solutions to matrix spike samples and LCSW (refer to Figure 3 of this SOP).
5. Add 1.5 mL (per 50 mL final volume) concentrated HNO_3 to sample.
6. Cover with a ribbed watch glass.
7. Place on heating device (hotplate or block digester) and evaporate to 10 - 15 mL.
8. Cool sample and add another 1.5 mL (per 50 mL final volume) concentrated HNO_3.
9. Resume heating until gentle reflux action occurs.
10. Continue heating, adding additional HNO_3 as necessary until digestion is complete.
11. Evaporate to 10 - 15 mL.
12. Cool sample and add 5 mL (per 50 mL final volume) 1:1 HCl. Resume heating and reflux gently for 15 minutes.
13. Cool sample and filter (if necessary) or decant into a graduated polyetheyne digestion tube. Rinse beaker with reagent water and filter or decant rinsate into specimen container.
14. Dilute to appropriate final volume with reagent water.
15. Cap sample container and shake gently to mix.

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

EXAMPLE PAGE FROM METALS SAMPLE PREPARATION LOGBOOK

Katahdin Analytical Services, Inc. — **Metals Preparation Benchsheet**

Reagent Information:
JT Baker HNO3: H14024 JT Baker HCL: IT3907 KMG H2O2: N/A **Method:** 3010

LCS / Spike — **LCS/Spiking Information:**

☑	☑	LV. CLPP-SPK-1 (ID/Vol):	MS1404 / 0.05	mL	Hot Plate/Block ID : A
☑	☑	CLPP-SPK-INT1 (ID/Vol):	MW2021 / 0.5	mL	Start Time/Temp.: 930 / 95 °C
☑	☑	CLPP-SPK-INT2 (ID/Vol):	MW2209 / 0.5	mL	End Time/Temp. : 1500 / 95 °C
☑	☑	Uranium Spike (ID/Vol):	MS1594 / 0.005	mL	Thermometer ID/Pos.: ALC 8 / 1.4
☐	☐	CLPP-SPK-4 (ID/Vol):	N/A /	mL	

Fisher Filter Paper: 411672325 2

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LCSWAB011CW0	AB011CW0	0.05	L	0.05	L	AQ	IC	AJB	02/01/2010	N/A	N/A	N/A	N/A		
PBWAB011CW0	AB011CW0		L		L	AQ	IC	AJB	02/01/2010	N/A	N/A	N/A	N/A		
SD0405-001	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						A
SD0405-001P	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-001S	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-002	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-003	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-004	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0405-005	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0422-001	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						B
SD0423-001	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						A
SD0429-001	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						B
SD0429-002	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-001	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-002	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-003	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						
SD0455-004	AB011CW0		L		L	AQ	IC	AJB	02/01/2010						

AJB 2-1-10

Digestion performed by: AJB **On:** 2-1-10

Page: AB003 **Revision:** 00

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 2

PREPARATION OF MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 50 mL Final Volume (mL)
Laboratory Control Sample (LCSW) and Matrix Spike	CLPP-SPK-1	Inorganic Ventures	0.050
	CLPP-SPK-INT1	Lab Prepared (see below)	0.50
	CLPP-SPK-INT2	Lab Prepared (see below)	0.50
	1000 mg/L Uranium Standard	Inorganic Ventures	0.005

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
CLPP-SPK-INT1	1000 mg/L Se	High Purity Standards	1.0
	1000 mg/L As	High Purity Standards	1.0
	1000 mg/L Pb	High Purity Standards	1.0
	1000 mg/L Cd	High Purity Standards	2.5
	1000 mg/L Sb	High Purity Standards	1.0
	10000 mg/L K	High Purity Standards	10.0
	10000 mg/L Na	High Purity Standards	7.5
	10000 mg/L Mg	High Purity Standards	5.0
	10000 mg/L Ca	High Purity Standards	2.5
CLPP-SPK-INT2	2007ICS-1	Inorganic Ventures	10.0
	1000 mg/L Sr	High Purity Standards	5.0
	1000 mg/L Sn	High Purity Standards	5.0
	10000 mg/L Si	High Purity Standards	5.0

TITLE: **ACID DIGESTION OF AQUEOUS SAMPLES BY EPA METHOD 3010 FOR ICP AND ICP-MS ANALYSIS OF TOTAL OR DISSOLVED METALS**

FIGURE 3

ELEMENT CONCENTRATIONS IN MATRIX SPIKES, LABORATORY CONTROL SAMPLES, AND THEIR COMPONENT SPIKING SOLUTIONS FOR DIGESTION OF AQUEOUS SAMPLES BY METHOD 3010

Element	CONCENTRATION IN SOLUTION, mg/L							
	Matrix Spike	LCSW	CLPP-SPK-1	CLPP-SPK-4	CLPP-SPK-INT1	CLPP-SPK-INT2	2007 ICS-1	1000 mg/L U
Aluminum	2.000	2.000	2000					
Antimony	0.500	0.500		100	100			
Arsenic	0.500	0.500		4	10			
Barium	2.000	2.000	2000					
Beryllium	0.050	0.050	50					
Boron	0.500	0.500		50		50	500	
Cadmium	0.250	0.250		5	25			
Calcium	2.500	2.500			250			
Chromium	0.200	0.200	200					
Cobalt	0.500	0.500	500					
Copper	0.250	0.250	250					
Iron	1.000	1.000	1000					
Lead	0.500	0.500		2	10			
Magnesium	5.000	5.000			500			
Manganese	0.500	0.500	500					
Molybdenum	0.300	0.300		30		30	300	
Nickel	0.500	0.500	500					
Potassium	10.000	10.000			1000			
Selenium	0.500	0.500		5	50			
Silicon	5.230	5.230				523	230	
Silver	0.050	0.050	50					
Sodium	7.500	7.500			750			
Strontium	0.500	0.500		50		50		
Thallium	0.500	0.500		5	10			
Tin	0.500	0.500		50		50		
Titanium	1.000	1.000		100		100	1000	
Uranium	0.100	0.100						1000
Vanadium	0.500	0.500	500					
Zinc	0.500	0.500	500					

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Heather Henningsen

Review Date: 5-11-11

SOP Number: CA-6024-05

SOP Title: Acid digestion of aqueous samples by EPA method 3010 for ICP and ICP-MS analysis of total or dissolved metals.

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: Gary Brewer **Date:** 05/11/11

QAO Signature: Lesie Dimond **Date:** 05/11/11

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Nicholas Tillotson

Review Date: 1/18/12

SOP Number: CA604-3010 D16

SOP Title: SW3050B

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: K. Brewer **Date:** 02/02/12

QAO Signature: Leslie Dimond **Date:** 020912

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Nicholas Tillotson

Review Date: 1/22/13

SOP Number: CA-604

SOP Title: Acid digestion of aqueous samples by EPA method 3010 for ICP and ICP-MS

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: A. Brewer **Date:** 02/26/13

QAO Signature: Leslie Dimond **Date:** 022613

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

SOP Number: CA-627
Revision History
Cover Page
Page 1

TITLE: TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020

Prepared By: George Brewer Date: 03/01

Approved By:

Group Supervisor: George Brewer Date: 04/02/01

Operations Manager: Jol C. Burton Date: 3/29/01

QA Officer: Deborah J. Nadeau Date: 03·27·01

General Manager: Date: 04/03/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
01	Changed acid solution conc. changed Run ID naming convention added data reduction and reporting procedures updated standards tables (4-8) updated Table 10 in include ISIS configuration	LAD	02-16-05	02-16-05
02	Sect. 4.2 - changed tebing size sect. 5 - changed acid conc. Sect. 7 - major changes to reflect current practises including reporting data in the metals database. Sect. 8 - major changes updating acceptance criteria. Updated Tables 1, 3 → 8, 10 & 11	LAD	04/06	04/06
03	Updated Tables 4, 5 and 6 with current standards. Updated Table 1 with serial dilution, Post Digestion Matrix spike, MSA, ICS-A, ICS-AB and IDL minimum frequency or criteria. Updated Sect. 8 regarding client specific requirements.	LAD	07/07	07/07
04	Section 7.18-changed instrument identifier to reflect new instrument; section 8-changed acceptance criteria and ICSAB analyte list; Table 1-updated acceptance criteria and corrective action to QC. Table 3-added all analytes to list-removed "for information only" list.	LAD	04/08	04/08
05	Updates to reflect changes from 6020 to 6020A. Added Hardness by calculation attachment. Added LLOC requirement and criteria to Sect 8 and Table 1. Added criteria to analyze PQL Std. at beginning and END of run.	LAD	02/09	02/09

TITLE: TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
06	Sect. 8 and QC Tables - Added DoD QSM references and criteria. Section 10 - Added references. Tables 4 → 7 - Added information pertaining to CCV conc. change	LAD	08/09	08/09
07	Added Table 2 with DoD QSM ver. 4.1 QC requirements. Updated Section 4.1, Table 10 and Table 11 with new autosampler information.	LAD	04/10	04/10
08	Sect. 1.1 - Added definitions. Sect. 3 4.1, 4.2, 5.2 7.9, 7.10, 7.11, 7.16 and 8.7 - minor changes to reflect current practice. Sect. 9 - added MDL, LOD and LOQ information. Sect. 10 - Added, ~~edited references~~. Updated Tables edited references LAD 042512	LAD	04/12	04/12

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-627-08**, titled **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**
.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-627-08**, titled **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

Recipient: _____ Date: _____

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

1.0 SCOPE AND APPLICATION

Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub-ppb (ug/L) concentrations of a large number of elements in water samples and in waste extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-leachable) elements are required.

ICP-MS has been applied to the determination of over 60 elements in various matrices. Analytes for which EPA has demonstrated the acceptability Method 6020 in a multi-laboratory study on solid wastes are listed as “analytes” in Table 4. Instrument detection limits, sensitivities, and linear ranges will vary with the matrices, and operating conditions. If Method 6020 is used to determine any analyte not listed in Table 4, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the waste to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality.

An appropriate internal standard is required for each analyte determined by ICP-MS. Recommended internal standards are ^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, and ^{209}Bi. The lithium internal standard should have an enriched abundance of ^{6}Li, so that interference from lithium native to the sample is minimized. Other elements may need to be used as internal standards when samples contain significant amounts of the recommended internal standards.

1.1 Definitions:

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified reagent water used to verify calibration accuracy.

ICP-MS - Inductively Coupled Plasma Mass Spectrometry.

ICS - Interference Check Samples - Two standards (ICS-A and ICS-AB) used to verify the effectiveness of interference correction equations. Solution ICS-A contains only interferents (Al, Ca, Fe, Mg, Na, K, P, S, Mo, Ti, C, Cl) at high

concentrations; solution ICS-AB contains interferents at the same concentrations as well as analytes at low (20 ug/L) concentrations.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence.

Internal Standard - Pure analytes added to a sample, extract, or standard solution in known amounts and used to measure the relative responses of other method analytes that are components of the same sample or solution. Internal standards must be analytes that are not native to the sample.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

LDR - Linear Dynamic Range - The concentration range over which the instrument response to an analyte is linear.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.
LOQ – Limit of Quantitation.- The minimum concentration of a target analyte that produces a quantitative result within specified limits of precision and bias.

PB - Preparation Blank - Reagent water that has been brought through the sample preparation process.

Post-Digestion Spike - An aliquot of a sample to which a known amount of analyte has been added before analysis and after digestion, if digestion is required.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of, analysts experienced in ICP-MS analysis by USEPA Method 6020 who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-MS. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, "Personnel Training & Documentation of Capability".

It is the responsibility of all Katahdin technical personnel involved in ICP-MS analysis by USEPA Method 6020 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to oversee that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

Samples, sample digestates, standards, and other reagents used in ICP analysis may contain high concentrations of acids and toxic metals. Spilled samples and reagents should be cleaned up from instrument and laboratory surfaces immediately.

Liquid argon represents a potential cryogenic and suffocation hazard and safe handling procedures should be employed at all times when handling liquid argon

tanks and fittings. Safety glasses and cryogenic-resistant gloves should be worn when changing or adjusting argon tanks.

The Agilent 7500 ICP-MS spectrometer is safety-interlocked to prevent user exposure to harmful electrical voltages, radio frequency emissions, ultraviolet radiation, high temperatures, and other hazards. At no time should the operator attempt to disable these interlocks or operate the instrument if any safety interlock is suspected to be disabled

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention and waste minimization techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Program for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in ICP-MS spectrometry may contain high concentrations of acids and toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested samples and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Instrument lab. Further information regarding waste classification and disposal may be obtained by consulting Katahdin Analytical Environmental Health and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

2.1 Prior to analysis, samples that require total (“acid-leachable”) values must be digested using appropriate sample preparation methods (such as USEPA Methods 3005 - 3051).

2.2 USEPA Method 6020 describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled argon plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of a vacuum interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratios and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

3.0 INTERFERENCES

Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). The Agilent 7500 ChemStation data system is used to correct for these interferences. This involves determining the signal for another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences which could affect ICP-MS determinations have been identified. Examples include $ArCl^+$ ions on the As signal and MoO^+ ions on the cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotopic abundances from the literature, the most precise coefficients for an instrument must be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^+$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}Ar\ ^{35}Cl^+$ signal):

Corrected ^{75}As signal (using natural isotopic abundances for coefficient approximations) =

(m/z 75 signal) - (3.13) (m/z 77 signal) + (2.73) (m/z 82 signal),

where the final term adjusts for any selenium contribution at 77 m/z.

NOTE: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}Se^+$, (e.g., $^{81}BrH^+$ from bromine wastes or ^{82}Kr from krypton contamination in the Ar).
Similarly:

Corrected ^{114}Cd signal (using natural isotopic abundances for coefficient approximations) = (m/z 114 signal) - (0.027) (m/z 118 signal) - (1.63)(m/z 108 signal),

where last 2 terms adjust for any tin or MoO^+ contributions at m/z 114.

NOTE: Cadmium values will be biased low by this type of equation when $^{92}ZrO^+$ ions contribute at m/z 108. Also, use of m/z 111 for Cd is even subject to direct ($^{92}ZrOH^+$) ions and indirect ($^{90}ZrO^+$) additive interferences when Zr is present.

NOTE: As for the arsenic equation above, the coefficients in the Cd equation are only illustrative. The most appropriate coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<l percent) counting precision.

The interference correction equations that are used by this laboratory in performing USEPA Method 6020 are listed in Table 4. The accuracy of these types of equations is based upon

the constancy of the observed isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the "parent" ion have not been found to be reliable, e.g., oxide levels can vary. If a correction for an oxide ion is based upon the ratio of parent-to-oxide ion intensities, the correction must be adjusted for the degree of oxide formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferent. This type of correction has been reported for oxide-ion corrections using ThO^+/Th^+ for the determination of rare earth elements. The use of aerosol desolvation and/or mixed plasmas have been shown to greatly reduce molecular interferences (the Agilent 7500 ICP-MS spectrometer employs spray chamber cooling to effect aerosol desolvation). These techniques can be used provided that method detection limit, accuracy, and precision requirements for analysis of the samples can be met.

3.1 Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000 mg/L) are recommended to minimize solid deposition. An internal standard can be used to correct for physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by matrix changes. The internal standard used should differ from the analyte of interest by no more than 50 amu. See table 14 for a list of internal standards used. When the intensity level of an internal standard is less than 70 percent or greater than 120 percent of the intensity of the first standard used during calibration, the sample must be reanalyzed after a fivefold (1+4) or greater dilution has been performed.

3.2 Memory interferences can occur when there are large concentration differences between samples or standards that are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences that are observed. The rinse period between samples must be long enough to eliminate significant memory interference.

4.0 APPARATUS AND MATERIALS

4.1 Agilent 7500 ICP-MS system, consisting of the Agilent 7500 ICP-mass spectrometer and its controlling computer data station. The spectrometer is capable of providing resolution better than or equal to unit resolution at 10% peak height. The Agilent 7500 mass range of 2-260 amu exceeds the method requirement of 2- 240 amu. The Agilent 7500 ChemStation software allows automatic corrections for isobaric interferences and correction for internal standard responses as required by the method. All critical argon flows including nebulizer argon are under mass flow

controller control and a peristaltic pump is used for sample introduction. Peripheral equipment includes a Elemental Scientific SC-4 PX Fast Autosampler and Sample Introduction system, and Bullzip PDF printer set to print to file ICPMS_CP.pdf located in folder PDF_PRINTS on the desktop.

4.2 Peristaltic pump tubing – 2-stop ESI PVC flared black-black (0.76 mm ID) and orange-green (0.38 mm ID). 3-stop Pharmed blue-yellow (1.52 mm ID).

4.3 15 ml 17x100 mm polypropylene or polystyrene disposable test tubes for samples and 50 ml polypropylene centrifuge tubes for standards.

4.4 Automatic adjustable-volume pipetters of suitable precision and accuracy. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Trace metal grade pipette tips.

4.6 Volumetric glassware or plasticware of suitable precision and accuracy.

4.7 Talc free vinyl gloves.

4.8 Argon gas supply (high purity grade gas or liquid, 99.99%).

4.9 For the determination of trace levels of elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment from dust etc. A clean laboratory work area, designed for trace element sample handling must be used. Standards, samples and blanks should be exposed to the laboratory environment as little as possible. The use of preparation blanks and spikes should be used to verify the absence of sources of contamination and loss. If necessary, polypropylene sample tubes should be rinsed and stored in dilute acid prior to use.

NOTE: Chromic acid must not be used for cleaning glassware for trace metals analysis.

5.0 REAGENTS

5.1 Acids used in the preparation of standards and for sample processing must be of high purity. Redistilled acids are recommended because of the high sensitivity of ICP-MS. Mallincrodt/Baker “Instra-Analyzed” trace-metals grade acids are appropriate. It is important to match the acid concentration in standards and samples. Concentrations of antimony and silver between 50-500 ug/L require 1% (v/v) HCl for stability; for concentrations above 500 ug/L additional HCl will be needed. For this reason, it is recommended that antimony and silver concentrations in samples and standards be maintained below 500 ppb wherever possible. Acids

are received in poly-coated glass bottles, and are stored under the hood in the Metals sample preparation laboratory at room temperature until use. All acids are considered to have a shelf life of three years from date of receipt unless otherwise indicated by the vendor. Refer to the current revision of Katahdin SOP CA-105, Reagent and Solvent Handling, for further details.

5.2 Laboratory reagent grade water, trace metals free, equivalent to ASTM Type 1 (ASTM D 1193), >18 Megohm/centimeter resistivity.

5.3 Single element and multielement stock standard solutions – purchased standards prepared from high purity salts or metals, and supplied by the vendors with certificates of purity and analysis. Refer to Tables 5 and 6 for a listing of stock standards required, and to Table 9 for element concentrations in stock standards. Purchased stock standards are received in polyethylene containers and are stored in their original containers at room temperature in the Metals Standards Preparation Laboratory. All purchased stock standards are given an expiration date as indicated by the manufacturer. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.4 Intermediate standard solutions – laboratory-prepared multielement standards that are used in the subsequent preparation of working standards. Refer to Table 6 for a listing of intermediate standards required and for preparation instructions. Refer to Table 8 for element concentrations in intermediate standards. Intermediate standards are stored at room temperature in acid-washed polyethylene containers in the Metals Standards Preparation Laboratory. Intermediate standards are assigned an expiration date of three months from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.5 Working standard solutions – laboratory-prepared multielement standards that are used to calibrate the instrument, to provide internal standardization through on-line addition, and to perform all necessary QC checks. Refer to Table 5 for a listing of working standards and for preparation instructions. Refer to Table 7 for element concentrations in working standards. Working standards are stored at room temperature in acid-washed polyethylene containers in the Metals Standards Preparation Laboratory. All working standards except the ICSA and ICSAB solutions are assigned an expiration date of three months from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. The ICSA and ICSAB solutions are assigned an expiration date of one week from the date of preparation, or the earliest expiration date of a component standard, whichever comes first. Refer to the current revision of Katahdin SOP CA-106, Standard Preparation, Documentation and Traceability, for further details.

5.6 Blanks: Three types of blanks are required for the analysis. The calibration blank is used in establishing the calibration curve. The preparation blank is used to monitor

for possible contamination resulting from the sample preparation procedure. The rinse blank is used to flush the system between all samples and standards.

5.6.1 The calibration blank consists of the same concentrations of the same acid(s) used to prepare the final dilution of the analyte calibration solutions (currently 1% HNO_3 and 0.5% HCl, v/v, in laboratory reagent grade water). Use of HCl for antimony and silver is cited in Section 5.1.

5.6.2 The preparation blank must be carried through the complete preparation procedure and contain the same volumes of reagents as the associated digested sample solutions.

5.6.3 The rinse blank consists of 4% HNO_3 and 0.5% HCl,v/v, in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Samples to be analyzed for trace metals by ICP-MS should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	6 months
Aqueous (dissolved)	P, G	250 mL	Filter, HNO_3 to pH < 2	6 months
Solid	P, G	10 g	Cool, 4°C	6 months

[1] P = polyethylene or, G = glass

7.0 PROCEDURES

7.1 Instrument control and data acquisition are completely automated through the use of the Agilent Chemstation software. The main Chemstation screen is accessed by double-clicking the “ICP-MS Top” icon on the Windows desktop. Autosampler tables are edited by selecting “Edit Sample Log Table” from the Sequence menu in the Agilent Chemstation software. In the following discussion, software menu items that are to be selected are printed in boldface. The instrument operating conditions, acquisition parameters, acquisition masses, and internal standards for analysis USEPA Method 6020 are detailed in Table 11.

7.2 Turn on the argon supply at the tank and set the pressure to >700 kPa.

7.3 Turn on the water chiller/recirculator.

7.4 Verify that the exhaust hood is in operation.

7.5 Ensure that the internal standard solution bottle is adequately full. Consumption is approximately 2.5 mL/hour.

7.6 Verify that the rinse station reservoir has an adequate supply of reagent water.

7.7 Verify that the drain reservoir has adequate room to accept the day's drain waste. Empty the reservoir as necessary into an appropriate waste container (Waste Stream A) located in the Hazardous Waste Storage Area.

7.8 Inspect the peristaltic pump tubes for signs of flattening and wear, and replace them as necessary. Clamp the peristaltic pump tubes into the peristaltic pump.

7.9 Open the Chemstation software by double-clicking the "ICP-MS Top" icon. Initiate the plasma by selecting **Instrument>>Instrument Control>>Plasma>>Plasma On** and allow the instrument to aspirate calibration blank solution for at least 45 minutes. During this warm-up, select **Tune>>Sensitivity>>Start** to start the instrument scanning the mass range. Verify that the flow of sample and internal standard solutions through the uptake lines and into the nebulizer is free from pulsations by introducing an air bubble into each line and observing its progress. Adjust the pump clamp tension on each line to obtain a constant, pulse-free flow.

7.10 After the 45 minute warm-up, check the responses of masses 82 and 83 to insure minimal krypton intereference with selenium. Mass 83 response should be < 2000 counts per second. Then aspirate the Instrument Tune Solution (10 ppb Li, Y, Ce, Tl) and check the responses and RSDs at masses 7, 89, and 205.

7.11 Generate a tune report by selecting **Tune>>File>>Generate Report**. Evaluate the tune report against the tune specifications listed in Table 12. If the tune passes all specifications, proceed to step 7.14.

7.12 If the tune report indicates unacceptable instrument performance for any parameter, initiate an autotune by selecting **Tune>>Autotune>>Run**. The Chemstation software will attempt to tune the instrument to meet the tune specifications, and will generate a new tune report after autotuning. Evaluate the new tune report against the tune specifications listed in Table 12.

7.13 Repeat step 7.12 until all tune specifications have been met. File the final tune report.

7.14 Aspirate the P/A tuning solution (see Table6) and run a P/A autotune by selecting **Tune>>Tune>>P/A Factor>>Run**. This will calibrate the pulse and analog modes of the detector. File the P/A report with the Tune report.

7.15 Load the instrument analytical method and calibrations table for USEPA Method 6020 into memory by selecting **Methods>>Load>>K6020.M>>K6020.C**.

7.16 Edit the sequence template "K6020.S" to create an analytical sequence table listing all of the samples to be analyzed. To do this, select "Edit Sample Log Table" from the **Sequence** menu in the Agilent Chemstation software. Double-click **SMPL** from the menu at the top left. Fill in the sample table with sample IDs, vial numbers, analytical method (K6020.M for all samples), dilution factors, and failure actions. When the sample table is complete, select **Print** to print this table. Close the "Edit Sample Log Table" window. Save the sample log table under a new name by selecting **Save** under the **Sequence** menu and then typing the name.

7.17 Load the autosampler racks according to the analytical sequence printout.

7.18 Select **Sequence>>Load and Run Sequence**, and select the appropriate autosampler sequence table from the displayed list. Enter the analyst's initials in the Operator box. Change data file name to appropriate designation. The protocol for naming data files is as follows: the 1st character is a letter that identifies the instrument ("J" for the Agilent 75000 ICP-MS), the 2nd character is a letter that identifies the year of the run ("X" for 2007, "Y" for 2008, etc.), the 3rd character is a letter that identifies the month of the run ("A" for January, "B" for February, etc.), the 4th and 5th characters are numbers that identify the date of the run ("01" for the first day of the month, etc.), and the 6th character is a letter that sequentially identifies the run ("A" for the first run of the day on that instrument, "B" for the second run, etc.). For example, the run identified as "JYA16A" is the first run of the day that was performed on January 16, 2008, using the Agilent 7500 ICP-MS. Select **Run**. The instrument will analyze all samples in the order listed in the table. Analysis must proceed in the sequence described in Table 10 to ensure that all necessary quality control samples are analyzed at the appropriate frequencies. A minimum of three replicate scans is required for all standards and samples. Analysis always begins with the analysis of a calibration blank followed by at least three multielement calibration standards to calibrate the instrument. The system is flushed with rinse blank between each sample and standard, and each sample and standard is aspirated for at least one minute prior to the beginning of mass scanning.

7.19 Analysis continues with analysis of the initial calibration verification standard (ICV) and the initial calibration blank (ICB) to verify the accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.20 A practical quantitation limit standard (PQL) is analyzed at the beginning of the run to verify calibration accuracy at the reporting limit. Refer to Section 8 and Table 1 for additional information.

7.21 A continuing calibration verification standard (CCV) and a continuing calibration blank (CCB) must be analyzed at the beginning of the run, after every ten samples,

and at the end of the run to verify the continued accuracy of the calibration. Refer to Section 8 and Table 1 for additional information.

7.22 Interference check standard solutions ICS-A and ICS-AB must be analyzed at the beginning of each run and every 12 hours to verify the adequacy of interference corrections. Refer to Section 8 and Table 1 for additional information.

7.23 All sample analytical results for a particular element that are bracketed (preceded or followed) by failing results in a calibration verification sample (ICV, ICB, CCV, or CCB) for that element must not be reported, except as noted in Sections 8.5, 8.6, and 8.9. The sample must be reanalyzed for the element in question.

7.24 All samples that exceed the linear dynamic range must be diluted and reanalyzed.

7.25 If dilutions of digested samples are performed, the measured element concentrations must be multiplied by the dilution factor prior to reporting. This is accomplished automatically by entering the dilution factor in the sample log table prior to initiation of analysis.

7.26 If an element has more than one monitored isotope, examination of the concentration calculated for each isotope, or the isotope ratios, will provide useful information for the analyst in detecting a possible spectral interference. Consideration should therefore be given to both primary and secondary isotopes in the evaluation of the element concentration. In some cases, secondary isotopes may be less sensitive or more prone to interferences than the primary recommended isotopes, therefore differences between the results do not necessarily indicate a problem with data calculated for the primary isotopes. In the case of Pb, quantitation is based on the sum of isotopes 206, 207 and 208 to compensate for any variation in naturally occurring isotope ratios. This is accomplished through the use of the interference correction equation for lead.

7.27 Calculations, aqueous samples: Final element concentrations for aqueous samples are reported in units of micrograms per liter (ug/L). The reported concentrations are calculated from measured digestate concentrations as follows:

$$\text{Concentration (ug/L)} = \frac{MC \times DF \times FV}{IV}$$

where: MC = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Final digestate volume (L)
IV = Digested sample volume (L)

7.28 Calculations, solid samples: Final element concentrations for solid samples are reported in units of milligrams per kilogram (mg/kg) on a dry weight basis. The

reported concentrations are calculated from measured digestate concentrations as follows:

$$\text{Concentration (mg/kg dry weight)} = \frac{MC \times DF \times FV \times 100}{W \times S}$$

where: MC = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Final digestate volume (L)
W = Weight of digested wet sample (g)
S = Percent solids

DATA REDUCTION AND REPORTING

7.29 When the analytical run is completed, the analyst should print a run summary and create a data import file.

7.30 Follow these steps to print the run summary: Open the FileView program using the "FIVIEW" icon on the Windows Desktop. Select the data file of interest and move the required samples into the "Process List". Make sure the "Corrected Data" box is checked. Click the "Process" button. The data will be displayed in a spreadsheet format.

7.31 Select "Configure" from the top menu and "Sublists" from the displayed options. Select "Load Sublist" and then select "K6020" from this list of options. Make sure the "Enable Sublist" box is checked. Click the "OK" button. This will display only the analyte masses in the spreadsheet.

7.32 Select "Quant Info" from the top menu and "Quant Results" from the displayed options. This will display the data in concentration units. Click on the cell in the top left corner of the spreadsheet to highlight all data. Select "Tools" from the top menu and "Import Data into Spreadsheet Application" from the displayed options. Click the "Save" button. The data is transferred to a Microsoft Excel spreadsheet. Minimize this window and return to the FileView spreadsheet.

7.33 Select "Configure" from the top menu and "Sublists" from the displayed options. Select "Load Sublist" and then select "INTSTDS" from this list of options. Make sure the "Enable Sublist" box is checked. Click the "OK" button. This will display only the internal standard masses in the spreadsheet.

7.34 Select "Count Info" from the top menu and "Counts/sec" from this list of options. This will display the data in counts per second units. Click on the cell in the top left corner of the spreadsheet to highlight all data. Select "Tools" from the top menu and "Import Data into Spreadsheet Application" from the displayed options. Click the "Save" button. The data is transferred to a Microsoft Excel spreadsheet. Copy the internal standard names and results and paste into the analyte results Excel spreadsheet.

7.35 In a separate cell of the Excel spreadsheet, input a formula to calculate the percent recovery of the internal standard relative to that of the calibration blank for each internal standard measurement of each sample.

7.36 Input a formula into the sample results cells to correct the data for dilution.

7.37 Format, print and save (as FileName.XLS, e.g. JYA28A.XLS) this run summary.

7.38 Follow these steps to create the data import file: Open the FileViewNT program using the "FIVIEWNT" icon on the Windows Desktop. Select the data file of interest and move the required samples into the "Process List". Make sure the "Corrected Data" box is checked. Click the "Process" button. The data will be displayed in a spreadsheet format.

7.39 Select "Configure" from the top menu and "Sublists" from the displayed options. Select "Load Sublist" and then select "K6020" from this list of options. Make sure the "Enable Sublist" box is checked. Click the "OK" button. This will display only the analyte masses in the spreadsheet.

7.40 Select "Quant Info" from the top menu and "Quant Results" from the displayed options. This will display the data in concentration units.

7.41 Select the "Transpose" from the menu.

7.42 Select "Tools" from the top menu and "Copy Selected Data to CSV File" from this list of options. Set the name the file as "FileName.CSV", e.g., "JYA28A.CSV". Click the "Save" button.

To import data into the Metals Database:

7.43 Select the "ICPMS Import" icon from the Windows Desktop, the ICPMS Import window will appear. Enter the datafile name without extension, (e.g., "JYA28A") and click the "Import" button.

7.44 When the "Import finished" message appears, close the ICPMS Import window and select the "KIMS_METALS" icon from the Windows Desktop. The Metals Database Main Menu will appear.

7.45 Select the "Reporting Menu" button. From the Reporting Menu screen select the Batch QC Menu button and then the "Calculate Batch QC" button.

7.46 From the resulting list of QC results, deselect any items that fail run QC or do not need ICP-MS analysis. Click on the "Accept Selected Batch QC" button.

7.47 From the Metals Main Menu, select the "Additional Data Handling" button. The Data Menu will appear. Select the "Report Added Compounds" button.

7.48 From the resulting list of sample results, deselect any items that fail run QC or do not need ICP-MS analysis. Click on the "Accept Data" button.

7.49 Once all associated data from an analysis run have been processed, go to the Generate Coverage portion of the Export Menu and print the Run Log and Logbook Page for the analysis run.

7.50 Combine these reports with the raw data printout and the Run Info Sheet and scan into a PDF format. Save in the "ICPMS DATA" section of the "METPDF" directory on the IMAGESERVER.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 6020 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgements. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases "qualified" data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in this section and in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument. This determination requires seven replicate analyses of a reagent blank, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on

performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed by each method on each instrument. This determination requires at least seven replicate digestions and analyses of reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the seven replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 A Lower Limit of Quantitation Check (LLQC) sample must be prepared and analyzed annually or on an as-needed basis to confirm the laboratory's Practical Quantitation Limits (PQLs). The LLQC sample is equivalent to the PQL standard (Section 8.9) but is carried through the entire sample preparation and analysis process. Element recoveries for the LLQC sample must fall within 70% to 130% of the expected concentrations to confirm the previously established PQLs.

ANALYTICAL RUN QC SAMPLES

8.4 Initial instrument calibration: The instrument is calibrated by running a calibration blank and at least three multielement calibration standards. For each element, calibration is performed fitting a single order equation to the calibration data, as follows:

$$Y=aX + [Blank]$$

where: Y = Concentration (ug/L)
X = Measured signal intensity (counts per second)
a = Slope of the calibration line
[Blank] = Measured signal intensity of the calibration blank

Fitting the calibration equation through the measured intensity of the calibration blank, rather than through the y-intercept of the line, provides the best calibration accuracy at the low end of the calibration range. When this equation is used, however, the Agilent software does not calculate a calibration coefficient. For this reason, calibration accuracy at the high end of the calibration range is checked by reanalyzing the highest calibration standard as a sample immediately after instrument calibration. Recoveries for all elements must be within 90% to 110% of the true value in the high calibration standard. If the high calibration standard fails, result for the failing elements may not be reported from the run until the problem is corrected and a passing high calibration standard has been analyzed. Calibration accuracy in the middle of the calibration range is verified by analysis of the CCV solution (see Section 8.6 below). Calibration accuracy at the reporting limit is verified by analysis of the PQL Check Standard (see Section 8.7 below).

8.5 The intensities of all internal standards must be monitored for every analysis. When the intensity of any internal standard fails to fall between 70 and 120 percent of the intensity of that internal standard in the initial calibration standard, the following procedure is followed. The sample must be diluted fivefold (1+4) and reanalyzed with the addition of appropriate amounts of internal standards. This procedure must be repeated until the internal standard intensities fall within the prescribed window. The intensity levels of the internal standards for the calibration blanks (ICB and CCBs) and calibration verification standards (ICV and CCVs) must agree within ± 20 percent of the intensity level of the internal standard of the original calibration solution. If they do not agree, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples.

8.6 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared by combining compatible elements from standard sources different than those of the calibration standards and at concentrations within the linear working range of the instrument. The results of the ICV must fall within 90% to 110% of the expected values. If the ICV fails, result for the failing elements may not be reported from the run, unless the ICV recovery is greater than 110% and the sample result is less than the PQL.

8.7 Continuing Calibration Verification (CCV) solutions are analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standards used for calibration at concentrations near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected values. If a CCV fails, results for the failing elements in samples bracketed by the failing CCV may not be reported, unless the CCV recovery is greater that 110% and the sample result is less that the PQL. For DoD analyses, results may not be reported without a valid CCV or report flagged results if reanalysis is not possible.

8.8 A Practical Quantitation Limit (PQL) Check Standard or low level continuing calibration verification (LLCCV) is analyzed at the beginning of each run (after the ICV and ICB samples) and at the end of each run. Element concentrations in this solution are one-fifth the laboratory's practical quantitation limit (assuming a 5-fold dilution of all digestates during analysis). Element recoveries for the PQL Check Standard must fall within 70% to 130% of the expected values (unless the samples analyzed are for the Department of Defense (80% to 120% recovery limits) or other client-specific limits are imposed). If the PQL Check Standard fails, results for the failing elements may not be reported from the run, unless the PQL Check Standard recovery is greater than the high limit and the sample result is less than the PQL.

8.9 A calibration blank solution is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than

the applicable reporting limit (or PQL) for each element. The reporting limit should be determined on a project specific basis, taking into account the data quality objectives for the project. This information must be communicated through a project QAPP and through the Katahdin project manager. When no project specific reporting limit is specified, the laboratory PQL shall be used. Some project specific limits may require reporting down to the MDL or IDL and taking corrective action based on these levels. Results that fall between the PQL and the IDL or MDL must always be flagged as "estimated" with a "J".

8.10 If an ICB or a CCB fails the acceptance criteria of less than the reporting limit, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed, with the following exception. If the result for an ICB or CCB is greater than the PQL (or reporting limit), sample results that are less than the PQL (or reporting limit) or that are greater than or equal to ten times the measured ICB or CCB concentration may be reported. Also, for failing elements, all samples analyzed after the last passing CCB must be reanalyzed, with the exception noted above.

8.11 To obtain analyte data of known quality, it is necessary to measure more than the analytes of interest in order to apply corrections or to determine whether interference corrections are necessary. If the concentrations of interference sources (such as C, Cl, Mo, Zr, W) are such that, at the correction factor, the analyte is less than the limit of quantitation and the concentration of interferents are insignificant, then the data may go uncorrected. Note that monitoring the interference sources does not necessarily require monitoring the interferent itself, but that a molecular species may be monitored to indicate the presence of the interferent. When correction equations are used, all QC criteria must also be met. Extensive QC for interference corrections are required at all times. The monitored masses must include those elements whose hydrogen, oxygen, hydroxyl, chlorine, nitrogen, carbon and sulfur molecular ions could impact the analytes of interest. Interference check solutions ICS-A and ICS-AB are analyzed at the beginning of each run and at least every 12 hours during the run to verify the effectiveness of interference corrections. Solution ICS-A contains high concentrations of interferents (Al, Ca, Fe, Mg, Na, P, K, S, C, Cl, Mo, and Ti) only. These should recover between 80% and 120% of the true value. The measured concentrations of other elements in this solution should be very low, indicating that interfering mass correction equations are adequate. Solution ICS-AB contains interferents at the same high concentrations, and all other analytes at 20 ug/L. Measured recoveries for all analytes should be within 80% to 120% of the true values.

PREPARATION BATCH QC SAMPLES

Each digestion batch of twenty or fewer samples will contain a preparation blank and a laboratory control sample. Each batch will also contain one or more of the following QC samples: laboratory control sample duplicate, sample duplicate, matrix spiked sample, or matrix spiked sample duplicate.

8.12 A preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) (or project specific reporting limit, if applicable) for each element. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL or reporting limit, associated sample results that are less than the PQL or reporting limit or that are greater than or equal to ten times the measured preparation blank concentration may be reported.

8.13 A laboratory control sample (LCSW, LCSO, or LCSS), consisting of spiked reagent water or a solid reference material carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless vendor-supplied limits (for solid reference materials) or laboratory-generated statistical limits are available. If a laboratory control sample fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the recovery of a laboratory control sample is greater than 120%, associated sample results that are less than the PQL or reporting limit may be reported.

SAMPLE MATRIX QC SAMPLES

8.14 The relative percent difference (RPD) between matrix duplicate, matrix spike duplicate, or laboratory control duplicate sample results is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = First sample or LCS result
D_2= Second (duplicate) sample or LCS result

A control limit of 20% RPD is applied to duplicate analysis, if the result is greater than 100 times the instrument detection limit. If the matrix duplicate analysis fails, the associated sample result must be flagged on the report of analysis.

8.15 The recovery for each element in a spiked sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If a recovery fails, the associated sample result must be flagged on the report of analysis. The spike recovery should be calculated as follows:

$$\text{Recovery (\%)} = \frac{S-U}{SA} * 100\%$$

where: S = Measured concentration of spiked aliquot
U = Measured concentration of unspiked aliquot
SA = Amount of spike added

8.16 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL), the measured concentration of a five-fold dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$\text{Difference (\%)} = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The action taken is dependent upon project requirements. The associated sample result may be flagged on the report of analysis, the sample may be reanalyzed at dilution to eliminate the interference, or a post-digestion spike may be performed (see section 8.16).

8.17 An analyte spike that is added to an aliquot of a prepared sample, or its dilution, should be recovered within 80% to 120% of the known value if the result for the unspiked aliquot is less than four times the amount of spike added. If the post-digestion spike is not recovered within these limits, the sample should be diluted and reanalyzed to compensate for the matrix interference or the method standard additions may be employed.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Detection (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revisions of USEPA Method 6020 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 6020A.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Current Version.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision

Agilent 7500 ICP-MS ChemStation Operator's Manual, Agilent Technologies, Inc., 2000.

List of Tables

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 1

QC REQUIREMENTS

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration, minimum 3 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient (r) $\geq$ 0.998	Recalibrate
Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within $\pm$ 10% of true value.	Do not use results for failing elements, unless ICV >110% and sample result < PQL/reporting limit.
Initial Calibration Blank (ICB)	Immediately after the ICV.	Absolute value of ICB < PQL or project specific reporting limit.	Do not use results if sample $\geq$ PQL/reporting limit and < 10x ICB level.
PQL Standard or LLCCV	At beginning and end of run	70-130% of true value	Do not use results for failing elements, unless PQL rec.> upper limit and sample result < PQL/reporting limit.
Continuing Calibration Verification (CCV)	At beginning of run, after every 10 samples, and at end of run.	Recovery within $\pm$ 10% of true value.	1) Do not use bracketed sample results for failing elements, unless CCV >110% and sample result < PQL/reporting limit. 2) Investigate and correct problem.
Continuing Calibration Blank (CCB)	Immediately after every CCV	Absolute value of CCB < PQL or project specific reporting limit.	Do not use sample results if $\geq$ PQL/reporting limit and < 10x CCB level.
Interference Check Solution A (ICS-A)	Before analyzing samples, and every 12 hours during a run.	Interferents: Recovery within $\pm$ 20% of true value. Analytes: No criteria established (Project specific criteria may apply)	Do not use sample results for failing elements.
Interference Check Solution AB (ICS-AB)	Before analyzing samples, and every 12 hours during a run.	Recovery within $\pm$ 20% of true value.	Do not use sample results for failing elements, unless ICSAB >120% and sample result < PQL/reporting limit.
Preparation Blank (PBW/PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL (standard practice), or based on the project specific guidelines.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration $\geq$ PQL and <10x the blank conc.
Laboratory Control Sample (LCSW/LCSS/LCSO)	At least one per digestion batch of 20 or fewer samples.	Recovery within $\pm$ 20% of true value, unless vendor-supplied or statistical limits have been established.	1) Investigate source of problem. 2) Redigest and reanalyze all associated samples, unless LCS >120% and sample result < PQL.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 1 (continued)

QC REQUIREMENTS

QC Sample	Minimum Frequency	Acceptance Criteria	Corrective Action
Duplicate Sample (D), Matrix Spike Duplicate (P), or LCS Duplicate (LC2W/LC2S/LC2O)	See section 8.11	1) RPD ≤ 20%, if sample > 100x IDL.	Flag results
Post-Digestion Matrix Spike (A)	When serial dilution fails and analyte concentration < 100 x MDL.	Recovery ± 20% of true value, if sample < 4x spike added.	Flag results and/or analyze sample by method of standard additions.
Serial Dilution (L)	1 per digestion batch	If original sample result is at least 50x IDL, 5-fold dilution must agree within ± 10% of the original result.	Flag result or dilute and reanalyze sample to eliminate interference.
Internal Standard (IS)	Appropriate IS required for all analytes in all samples. Mass of IS must be <50 amu different from that of analyte.	1) For each sample, IS intensity within 70%-120% of that of initial calib. blank. 2) For ICV, ICB, CCV, and CCB, IS intensity within 80%-120% of that in initial calib. blank.	Do not use results for failing elements.
Instrument Detection Limit (IDL) Study	Quarterly.	IDL < MDL PQL at least 2-3x IDL	1) Repeat IDL study. 2) Raise PQL.
Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
Lower Limit of Quantitation Check (LLQC) Sample	Digest and analyze annually or as needed to confirm PQLs	70% - 130% of true value	Reevaluate PQLs
Method of Standard Additions	When matrix interference is suspected	r ≥ 0.995	Dilute and reanalyze sample to eliminate interference.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Refer to current revision of SOP QA-806				
LOQ establishment and verification	Refer to current revision of SOP QA-806				
Instrument detection limit (IDL) study	At initial set-up and after significant change in instrument type, personnel, test method, or sample matrix.	IDLs shall be ≤ LOD.	NA.	NA.	Samples may not be analyzed without a valid IDL.
Tuning	Prior to ICAL.	Mass calibration ≤ 0.1 amu from the true value; Resolution < 0.9 amu full width at 10% peak height; For stability, RSD ≤ 5% for at least four replicate analyses.	Retune instrument then reanalyze tuning solutions.	Flagging criteria are not appropriate.	No analysis shall be performed without a valid MS tune.
Initial calibration (ICAL) for all analytes (minimum one high standard and a calibration blank)	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, r ≥ 0.995.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analytes within ± 10% of true value.	Verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.

TABLE 2 (cont)

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All analytes within ± 10% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Low-level calibration check standard	Daily, after one-point ICAL.	Within ± 20% of true value.	Correct problem, then reanalyze.	Flagging criteria are not appropriate.	No samples may be analyzed without a valid low-level calibration check standard. Low-level calibration check standard should be less than or equal to the reporting limit.
Linear dynamic range or high-level check standard	Every 6 months.	Within ±10% of true value.	NA.	NA.	
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). For negative blanks, absolute value < LOD. Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD. For negative blanks, absolute value < LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
Interference check solutions (ICS-A and ICS-AB)	At the beginning of an analytical run and every 12 hours.	ICS-A: Absolute value of concentration for all non-spiked analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value. May use < LOD for some projects.	Terminate analysis, locate and correct problem, reanalyze ICS, reanalyze all samples.	If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS.	

TABLE 2 (cont)

DoD QSM REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
LCS containing all analytes to be reported	One per preparatory batch.	Water: Recovery must be within + 20% of the true value Soil: Recovery must be within vendor supplied limits (varies by lot).	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix spike (MS)	One per preparatory batch per matrix	For matrix evaluation, use recovery must be within + 20% of the true value.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests (dilution test and post-digestion spike addition) are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix.	MSD: For matrix evaluation use recovery must be within + 20% of the true value. MSD or sample duplicate: RPD < 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.
Dilution test	One per preparatory batch.	For samples with concentrations > 50 x LOQ then five-fold dilution must agree within ± 10% of the original measurement.	Perform post-digestion spike addition.	Flagging criteria are not appropriate.	Only applicable for samples with concentrations > 50 x LOQ.
Post digestion spike addition	When dilution test fails or analyte concentration for all samples < 50 x LOD.	Recovery within 75-125%	Run all associated samples in the preparatory batch by method of standard additions (MSA) or see flagging criteria.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	Spike addition should produce a concentration of 10 – 100 x LOQ.
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Internal standards (IS)	Every sample.	IS intensity within 30-120% of intensity of the IS in the ICAL.	Flagging criteria are not appropriate.	Reanalyze sample at 5-fold dilution with addition of appropriate amounts of internal standards.	
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-627-07	METHOD 6020, current revision
Apparatus/Materials		
Reagents		
Sample preservation/ handling		
Procedures		
QC - Spikes		
QC - LCS		
QC - Accuracy/Precision		
QC - MDL		
QC - Calibration Blanks	Acceptance criteria employed for 6020: ± PQL	Acceptance criteria stated in 6020: <10% of PQL

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 4

ISOTOPES MONITORED AND CORRECTION EQUATIONS USED FOR USEPA METHOD 6020

Element Class	Element	Sym-bol	Isotopes Monitored	Correction Equations [See note 1]
Analytes	Aluminum	Al	27	
	Antimony	Sb	121, 123	
	Arsenic	As	75	^{75}As = (75)*1 - (77)*2.95 + (82)*2.548 – (83)*2.571 [See note 2]
	Barium	Ba	135, 137	
	Beryllium	Be	9	
	Boron	B	11	
	Cadmium	Cd	106, 108, 111, 114	^{111}Cd = (111)*1 – (108)*1.073 + (106)*0.764 [See note 3] ^{114}Cd = (114)*1 – (118)*0.0268 [See note 4]
	Calcium	Ca	44	^{44}Ca = (44)*1 – (88)*0.0169 [See note 7]
	Chromium	Cr	52, 53	
	Cobalt	Co	59	
	Copper	Cu	63, 65	
	Iron	Fe	54, 56, 57	^{54}Fe = (54)*1 – (52)*0.0282 [See note 8] ^{57}Fe = (57)*1 – (43)*0.03 [See note 9]
	Lead	Pb	206, 207, 208	^{208}Pb = (208)*1 + (206)*1 + (207)*1 [See note 5]
	Magnesium	Mg	25	
	Manganese	Mn	55	
	Molybdenum	Mo	98	^{98}Mo = (98)*1 – (99)*0.146 [See note 10]
	Nickel	Ni	60, 61	
	Potassium	K	39	
	Selenium	Se	82	^{82}Se = (82)*1 – (83)*1.009 [See note 11]
	Silver	Ag	107, 109	
	Sodium	Na	23	
	Strontium	Sr	88	
	Thallium	Tl	203, 205	
	Thorium	Th	232	
	Tin	Sn	118, 120	
	Tungsten	W	182	
	Uranium	U	238	
	Vanadium	V	51	^{51}V = (51)*1 – (53)*2.95 + (52)*0.333 [See note 12]
	Zinc	Zn	66, 67, 68	
Internal Stan-dards.	Bismuth	Bi	209	
	Germanium	Ge	72	
	Indium	In	115	^{115}In = (115)*1 – (118)*0.016 [See note 6]
	Lithium	Li	6	
	Scandium	Sc	45	
	Terbium	Tb	159	
	Yttrium	Y	89	

TABLE 4 (continued)

ISOTOPES MONITORED AND CORRECTION EQUATIONS USED FOR USEPA METHOD 6020

Notes:

1) Numbers in parentheses, e.g "(51)", indicate measured counts at the indicated mass.
2) Corrects for ArCl interference, taking into account secondary interferences from Se and Kr
3) Corrects for MoO interference, taking into account secondary interference from ^{108}Cd
4) Corrects for Sn interference
5) Corrects for variations in isotopic composition of lead
6) Corrects for Sn interference
7) Corrects for interference from $^{88}Sr^{2+}$
8) Corrects for Cr interference
9) Corrects for Ca interference
10) Corrects for Ru interference
11) Corrects for Kr interference
12) Corrects for ClO, taking into account secondary interference from Cr

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 5

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Calibration Standard 1 (S1) (1.0% HNO_3 / 0.5% HCl)	CL-CAL-3	Spex Industries	0.005
	ICP-MS-MIX-Z	Lab Prepared	0.01
	ICP-MS CAL 1	Lab Prepared	0.025
Calibration Standard 2 (S2) (1.0% HNO_3 / 0.5% HCl)	CL-CAL-3	Spex Industries	0.05
	ICP-MS-MIX-Z	Lab Prepared	0.10
	ICP-MS CAL 1	Lab Prepared	0.25
Calibration Standard 3 (S3) / CCV (1.0% HNO_3 / 0.5% HCl)	CL-CAL-3	Spex Industries	0.25
	ICP-MS-MIX-Z	Lab Prepared	0.50
	ICP-MS CAL 1	Lab Prepared	1.25
Calibration Standard 4 (S4) / High Standard (1.0% HNO_3 / 0.5% HCl)	CL-CAL-3	Spex Industries	0.50
	ICP-MS-MIX-Z	Lab Prepared	1.0
	ICP-MS CAL 1	Lab Prepared	2.5
Initial Calibration Verification (ICV) (1.0% HNO_3 / 0.5% HCl)	CL-ICS-1,CL-ICS-4, CL-ICS-5	Spex Industries	0.20 of each
	CL-ICS-3	Spex Industries	2.0
	1000 mg/L Si	Inorganic Ventures	0.040
	1000 mg/L Al	Inorganic Ventures	0.038
	1000 mg/L B, W	Inorganic Ventures	0.002 of each
Continuing Calibration Verification (CCV) (1.0% HNO3/ 0.5% HCl)	CL-CAL-3	Spex Industries	0.25
	ICP-MS-MIX-Z, ICP-MS CAL 1, ICP-MS-MIX-Y	Lab Prepared	0.50 of each

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 5 (continued)

PREPARATION OF CALIBRATION AND QUALITY CONTROL STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
Practical Quantitation Limit Solution (PQL) (1.0% HNO_3 / 0.5% HCl)	ICP-MS PQL Intermediate	Lab Prepared	0.1
Interference Check Solution A (ICS-A) (1.0% HNO_3 / 0.5% HCl)	6020ICS-0A	Inorganic Ventures	10.0
Interference Check Solution AB (ICS-AB) (1.0% HNO_3 / 0.5% HCl)	6020ICS-0A	Inorganic Ventures	10.0
	ICP-MS-CAL 1	Lab Prepared	1.0
	ICP-MS ICSAB Intermediate	Lab Prepared	1.0
P/A Tuning Solution (1.0% HNO_3 / 0.5% HCl)	1000 mg/L Co, Cr, Mo, Mn, Pb, Sb, Sr, U, V	High Purity Standards	0.02
	10,000 mg/L Al, K, Na	High Purity Standards	0.002
Instrument Tuning Solution (1.0% HNO_3 / 0.5% HCl)	ICP-MS-TS-2	High Purity Standards	0.10
Internal Standard Solution (5.0% HNO_3 / 0.5% HCl)	Internal Standard Mix	Spex Industries	10
Method Tuning Solution (1.0% HNO_3 / 0.5% HCl)	ICP-MS Method Tune Intermediate	Lab Prepared	1.0
	Internal Standard Mix 1	Spex Industries	1.0

TABLE 6

PREPARATION OF INTERMEDIATE STANDARDS

Sample or Solution Name	Component Solution Name	Source of Component	Amount of Component Added per 100 mL Final Volume (mL)
ICP-MS PQL Intermediate (1.0% HNO_3 / 0.5% HCl)	10,000 mg/L K, Na	High Purity Standards or Inorganic Ventures	2.0 of each
	10,000 mg/L Si	High Purity Standards or Inorganic Ventures	1.0
	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.60
	1000 mg/L B	High Purity Standards or Inorganic Ventures	0.40
	10,000 mg/L Ca, Fe, Mg 1000 mg/L Zn	High Purity Standards	0.20 of each
	1000 mg/L As, Se, V, W, Sr, Sn, Mo, Cr	High Purity Standards or Inorganic Ventures	0.10 of each
	1000 mg/L Cu	High Purity Standards	0.06
	1000 mg/L Ba, Mn, Ni	High Purity Standards	0.04 of each
	1000 mg/L U, Be, Cd, Co, Ag, Tl, Pb, Sb	High Purity Standards	0.02 of each
ICP-MS CAL 1 (1.0% HNO_3 / 0.5% HCl)	1000 mg/L Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, U, V, Zn	High Purity Standards	0.2 of each
	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.02
ICP-MS-MIX-Z (1.0% HNO_3 / 0.5% HCl)	10,000 mg/L K, Na, Fe, Mg, Ca	High Purity Standards or Inorganic Ventures	5.0 of each
	10,000 mg/L Si	High Purity Standards or Inorganic Ventures	1.0
	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.95
	1000 mg/L B, Sn, Sr, W	High Purity Standards or Inorganic Ventures	0.50 of each

TABLE 6 (Cont'd)

PREPARATION OF INTERMEDIATE STANDARDS

ICP-MS-MIX-Y (1.0% HNO3/ 0.5% HCl)	10,000 mg/L Al	High Purity Standards or Inorganic Ventures	0.030
	1000 mg/L As, Ba, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, V, Zn	High Purity Standards or Inorganic Ventures	0.30 of each
ICP-MS ICSAB Intermediate (1.0% HNO_3 / 0.5% HCl)	10,000 mg/L Si	High Purity	0.50
	1,000 mg/L B, Sn, Sr, W	High Purity or Inorganic Ventures	0.20 each
ICP-MS Method Tune Intermediate (1.0% HNO_3 / 0.5% HCl)	1000 mg/L Be, Co, Pb, Tl 10,000 mg/L Mg	High Purity Standards or Inorganic Ventures	0.1 of each

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 7

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L							
	S1	S2	S3	S4	ICV	PQL	CCV	P/A Tune Soln.
Aluminum	10.0	100.0	500.0	1000.0	400.0	60.0	500.0	200
Antimony	0.5	5.0	25.0	50.0	20.0	0.2	25.0	200
Arsenic	0.5	5.0	25.0	50.0	20.0	1.0	25.0	
Barium	0.5	5.0	25.0	50.0	20.0	0.4	25.0	
Beryllium	0.5	5.0	25.0	50.0	20.0	0.2	10.0	
Boron	0.5	5.0	25.0	50.0	20.0	4.0	25.0	
Cadmium	0.5	5.0	25.0	50.0	20.0	0.2	10.0	
Calcium	100.0	1000.0	5000.0	10000.0	4000.0	20.0	5000.0	
Chromium	0.5	5.0	25.0	50.0	20.0	1.0	25.0	200
Cobalt	0.5	5.0	25.0	50.0	20.0	0.2	10.0	200
Copper	0.5	5.0	25.0	50.0	20.0	0.6	25.0	
Iron	100.0	1000.0	5000.0	10000.0	4000.0	20.0	5000.0	
Lead	0.5	5.0	25.0	50.0	20.0	0.2	25.0	200
Magnesium	100.0	1000.0	5000.0	10000.0	4000.0	20.0	5000.0	
Manganese	0.5	5.0	25.0	50.0	20.0	0.4	25.0	200
Molybdenum	0.5	5.0	25.0	50.0	40.0	1.0	25.0	200
Nickel	0.5	5.0	25.0	50.0	20.0	0.4	25.0	
Potassium	100.0	1000.0	5000.0	10000.0	4000.0	200.0	5000.0	200
Selenium	0.5	5.0	25.0	50.0	20.0	1.0	25.0	
Silicon	10.0	100.0	500.0	1000.0	400.0	100.0	500.0	
Silver	0.5	5.0	25.0	50.0	20.0	0.2	10.0	
Sodium	100.0	1000.0	5000.0	10000.0	4000.0	200.0	5000.0	200
Strontium	0.5	5.0	25.0	50.0	20.0	1.0	25.0	200
Thallium	0.5	5.0	25.0	50.0	20.0	0.2	10.0	
Tin	0.5	5.0	25.0	50.0	20.0	1.0	25.0	
Tungsten	0.5	5.0	25.0	50.0	20.0	1.0	25.0	
Uranium	0.5	5.0	25.0	50.0	20.0	0.2	10.0	200
Vanadium	0.5	5.0	25.0	50.0	20.0	1.0	25.0	200
Zinc	0.5	5.0	25.0	50.0	20.0	2.0	25.0	

TABLE 7 (continued)

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L				
	ICSA[1]	ICSAB[1]	Internal Std Solution	Method Tune Solution	Instrument Tuning Solution
Aluminum	100000	100000			
Antimony		20			
Arsenic		20			
Barium		20		10	
Beryllium		20			
Boron		20			
Cadmium		20			
Calcium	100000	100000			
Chromium		20			
Cobalt		20		10	
Copper		20			
Iron	100000	100000			
Lead		20		10	
Magnesium	100000	100000		100	
Manganese		20			
Molybdenum	2000	2000			
Nickel		20			
Potassium	100000	100000			
Selenium		20			
Silver		20			
Sodium	100000	100000			
Strontium		20			
Thallium		20		10	10.0
Tin		20			
Tungsten		20			
Uranium		20			
Vanadium		20			
Zinc		20			
Bismuth			1000.0	10	
Germanium			1000.0	10	
Indium				10	
Lithium (^{6}Li)			1000.0	10	
Scandium			1000.0	10	
Terbium			1000.0	10	
Yttrium			1000.0	10	10.0
Cerium					10.0

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 7 (continued)

ELEMENT CONCENTRATIONS IN WORKING STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L				
	ICSA[1]	ICSAB[1]	Internal Std Solution	Method Tune Solution	Instrument Tuning Solution
Lithium					10.0

1) Solution also contains 1000 mg/L Chloride, 200 mg/L Carbon, and 100 mg/L Phosphorus and Sulfur, and 2mg/L Titanium.

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 8

ELEMENT CONCENTRATIONS IN INTERMEDIATE STANDARDS

ELEMENT	CONCENTRATION IN SOLUTION, mg/L					
	MS-MIX-Z	ICP-MS PQL Intermediate	ICP-MS-MIX-Y	ICP-MS Method Tune Intermediate	ICP-MS CAL 1	ICP-MS ICSAB Intermediate
Aluminum	95.0	6.0	3.0		0.2	
Antimony		0.02	3.0		0.2	
Arsenic		0.10	3.0		0.2	
Barium		0.04	3.0		0.2	
Beryllium		0.02		1.0	0.2	
Boron	5.0	4.0				0.2
Cadmium		0.02			0.2	
Calcium	500	2.0				
Chromium		0.10	3.0		0.2	
Cobalt		0.02		1.0	0.2	
Copper		0.06	3.0		0.2	
Iron	500	2.0				
Lead		0.02	3.0	1.0	0.2	
Magnesium	500	2.0		10.0		
Manganese		0.04	3.0		0.2	
Molybdenum		0.10	3.0		0.2	
Nickel		0.04	3.0		0.2	
Potassium	500	20.0				
Selenium		0.10	3.0		0.2	
Silicon	100	10.0				5.0
Silver		0.02			0.2	
Sodium	500	20.0				
Strontium	5.0	0.10				0.2
Thallium		0.02		1.0	0.2	
Tin	5.0	0.10				0.2
Tungsten	5.0	0.10				0.2
Uranium		0.020			0.2	
Vanadium		0.10	3.0		0.2	
Zinc		0.20	3.0		0.2	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 9

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, mg/L				
	Instrument Calibration Standard 3 (Spex)	CL-ICS-1 (Spex)	CL-ICS-3 (Spex)	CL-ICS-4 (Spex)	CL-ICS-5 (Spex)
Aluminum		10.0			
Antimony		10.0			
Arsenic		10.0			
Barium		10.0			
Beryllium		10.0			
Boron					
Cadmium		10.0			
Calcium	1000		200.0		
Chromium		10.0			
Cobalt		10.0			
Copper		10.0			
Iron	1000		200.0		
Lead		10.0			
Magnesium	1000		200.0		
Manganese		10.0			
Molybdenum				10.0	10.0
Nickel		10.0			
Potassium	1000		200.0		
Selenium		10.0			
Silver		10.0			
Sodium	1000		200.0		
Strontium					10.0
Thallium		10.0			
Thorium				10.0	
Tin					10.0
Tungsten					
Uranium				10.0	
Vanadium		10.0			
Zinc		10.0			

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 9 (continued)

ELEMENT CONCENTRATIONS IN STOCK STANDARDS

Element	CONCENTRATION IN SOLUTION, ug/L		
	6020ICS-0A[1] (Inorganic Ventures)	**Internal Standard Mix 1 (Spex)**	**ICP-MS-TS-2 (High Purity)**
Aluminum	1000		
Arsenic			
Cadmium			
Calcium	1000		
Chromium			
Cobalt			
Copper			
Iron	1000		
Magnesium	1000		
Manganese			
Molybdenum	20.0		
Nickel			
Potassium	1000		
Silver			
Sodium	1000		
Zinc			
Bismuth		1000	
Cerium			10000
Germanium		1000	
Indium		1000	
Lithium			10000
Lithium (^{6}Li)		1000	
Scandium		1000	
Terbium		1000	
Thallium			10000
Yttrium		1000	10000

1) Solution also contains 10000 mg/L Chloride, 2000 mg/L Carbon, and 1000 mg/L Phosphorus and Sulfur, and 20 mg/L Titanium.

TABLE 10

REQUIRED ANALYTICAL SEQUENCE

Sequence Number	Standard/Sample	Purpose
1	Method Tuning Solution	Verify mass calibration and resolution
2	S0 (Calibration Blank)	Initial calibration
3	S1 (Calibration Standard)	Initial calibration
4	S2 (Calibration Standard)	Initial calibration
5	S3 (Calibration Standard)	Initial calibration
6	S4 (Calibration Standard)	Initial calibration
7	ICV (Initial Calibration Verification)	Check calibration accuracy
8	ICB (Initial Calibration Blank)	Check calibration accuracy
9	PQL (Practical Quantitation Limit)	Check calibration accuracy at low concentration
10	ICS-A (Interference Check Solution A)	Verify accuracy of mass correction equations
11	ICS-AB (Interference Check Solution AB)	Verify accuracy of mass correction equations
12	CCV (Continuing Calibration Verification)	Check calibration stability
13	CCB (Continuing Calibration Blank)	Check calibration stability
14-23	Analyze up to 10 samples	
24	CCV (Continuing Calibration Verification)	Check calibration stability
25	CCB (Continuing Calibration Blank)	Check calibration stability
…	Continue analyzing sequences of up to 10 samples, followed by a CCV and a CCB	
…	After last analytical sample, analyze PQL , followed by a CCV and a CCB	

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

TABLE 11

INSTRUMENT OPERATING CONDITIONS

Data Acquisition Program	Acquisition Mode	Spectrum
	Points per Mass	3
	Number of Replicates	3
	Detector Mode	Auto for all elements
	Integration Time per Point (for listed masses and their correction masses)	0.10 sec for Li, B, ^{29}Si, Sc, V, Cr, Mn, Ni, Cu, Zn, Y, Mo, Ag, In, Sn, Sb, Ba, Tb, W, Tl, Pb, Bi, Th, U
		0.30 sec for Be, As, Cd, Ge
		0.010 sec for Na, Al, K, ^{28}Si
		0.030 for Ca, Fe, Sr
		1.00 sec for Se
		0.050 sec for Mg, Co
	Spray Chamber Temperature	2^{o} C
	Total Acquisition Time	105 sec for 3 replicates
Peristaltic Pump Program	Analysis Speed	0.15 rps
Before Acquisition	Uptake Speed	0.15 rps
	Uptake Time	5 sec
	Stabilization Time	15 sec
After Acquisition (Probe Rinse)	Rinse Speed	0.15 rps
	Rinse Time (sample)	5 sec
	Rinse Time (standard)	5 sec
After Acquisition (Rinse)	Rinse Vial	1
	Uptake Speed	0
	Uptake Time	0 sec
	Stabilization Time	0 sec
Calibration Curve fit	All quantitation masses	Y=ax+(blank)
	All internal standard masses	(Excluded)
	All interference correction masses	(Excluded)
Reporting Parameters	QC Reports	On-Printer
	All Other Reports	Off

TABLE 12

INSTRUMENT TUNE SPECIFICATIONS

Sensitivity	Li >5000 cts/0.1 sec/10 ppb
	Y >10,000 cts/0.1 sec/10 ppb
	Tl >5000 cts/0.1 sec/10 ppb
Precision	Li <8% RSD (0.1 sec integration time)
	Y <5% RSD (0.1 sec integration time)
	Tl <5% RSD (0.1 sec integration time)
Oxides	<1.0%
Doubly Charged (Ce^{++}/Ce^{+})	<2.0%
Background	Li <15 cps
	Y <15 cps
	Tl <15 cps
Mass Resolution	Width at 10% peak height: 0.7-0.8 amu
Mass Axis	Li ±0.1 amu of nominal mass
	Y ±0.1 amu of nominal mass
	Tl ±0.1 amu of nominal mass

TABLE 13

METHOD TUNE SPECIFICATIONS

Precision	≤5% RSD of 4 replicates
Mass Resolution	Width at 10% peak height: <0.9 amu
Mass Calibration	±0.1 amu of nominal mass

TABLE 14

REPORTED ISOTOPES AND INTERNAL STANDARDS

ELEMENT	MASS	INTERNAL STANDARD (mass)
Aluminum	27	Scandium (45)
Antimony	123	Terbium (159)
Arsenic	75	Germanium (72)
Barium	135	Terbium (159)
Beryllium	9	Lithium (6)
Boron	11	Lithium (6)
Cadmium	114	Germanium (72)
Calcium	44	Scandium (45)
Chromium	52	Germanium (72)
Cobalt	59	Germanium (72)
Copper	65	Germanium (72)
Iron	57	Germanium (72)
Lead	208	Bismuth (209)
Magnesium	25	Scandium (45)
Manganese	55	Germanium (72)
Molybdenum	98	Germanium (72)
Nickel	60	Germanium (72)
Potassium	39	Scandium (45)
Selenium	82	Germanium (72)
Silicon	29	Scandium (45)
Silver	107	Germanium (72)
Sodium	23	Scandium (45)
Strontium	88	Germanium (72)
Thallium	203	Bismuth (209)
Thorium	232	Bismuth (209)
Tin	118	Terbium (159)
Tungsten	182	Terbium (159)
Uranium	238	Bismuth (209)
Vanadium	51	Germanium (72)
Zinc	66	Germanium (72)

TITLE: **TRACE METALS ANALYSIS BY ICP-MS USING USEPA METHOD 6020**

ATTACHMENT 1

HARDNESS BY CALCULATION

As referenced in "Standard Methods for the Examination if Water and Wastewater," Methods 2340 A & B, Hardness Introduction and Hardness by Calculation, American Public Health Association, 18th Edition, Revised 1992, total hardness is the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate, in milligrams per liter.

Once the calcium and magnesium concentrations have been determined by EPA methods 6010, 6020, 200.7 or 200.8, the total hardness of an aqueous sample may be calculated as follows:

$$\text{Total Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L) + 4.118\ (Mg, mg/L)$$

The calcium hardness of an aqueous sample may also be calculated as follows:

$$\text{Calcium Hardness, mg equivalent } CaCO_3/L = 2.497\ (Ca, mg/L)$$

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Prepared By: George Brewer Date: 01/01

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: Joel C. Burton Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1-29-01

General Manager: Dennis P. Hoffman Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
00 ~~7470A~~	NA	DH	1-29-01	1/29/01
01	Revised Sect. 4, 5 and 7 to reflect current practice. Revised Sect. 8 to reflect current QC limits. Revised sect. 10 to reflect current Applicable Documents and references. Removed figure 2. Update table 1 to reflect current QC limits. Minor changes throughout	LAD	02-16-05	02-16-05
02	Updated Fig. 1 - new prep logbook page	LAD	04/08	04/08
03	Updated Figure 1 - Example of a mercury Preparation Logbook page	LAD	03/09	03/09
04	Added LOD definition. Updated sections 8,9,10 and Table 1 for DOD QSM version 4.1 compliance.	DH	08/09	08/09

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
05	Added Table 2 - DoD QSM version 4.1 QC Requirements.	LAD	04/10	04/10
06	Sect. 4.4 - Changed thermometer type. Sect. 7.3 - Changed type of marker used. Table 1 - Added PQL standard corrective action. Table 2 - added comments for calibration blank. Sect. 9 - Added MDL, LOD and LOQ information	LAD	05/11	05/11
07	Sect. 7 - Calibration prep from digesting all to digesting high STD. and diluting down. Added serial dilution and PDS to sect. 8. Added more MDL, LOD & LOQ information to Sect. 9. Updated and added references to Sect. 10	LAD	04/12	04/12

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document **SOP CA-615-07,** titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470.**

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document **SOP CA-615-07,** titled **DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470.**

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TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services, Inc. personnel for the digestion and analysis aqueous samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in groundwaters, aqueous wastes, and mobility-procedure extracts under USEPA Method 7470 (Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods, SW-846, 2nd edition, 1982 (revised 1984), 3rd edition, 1986, and Updates I, II, IIA, and III 1996, Office of Solid Waste and Emergency Response, U.S. EPA.

1.1 Definitions

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy periodically during analysis.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.
ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory grade reagent water used to verify calibration accuracy.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

PB - Preparation Blank - Laboratory grade reagent water that has been brought through the sample preparation process.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

Serial Dilution - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7470. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7470 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, that their work is properly documented, and to indicate periodic review of the associated logbooks.

1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Rubber gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this

method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with the Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Management Plan and follow appropriate procedures such as wearing safety glasses and gloves when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location and use of all safety equipment.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address their waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Hazardous Waste Management Plan and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

The cold vapor atomic absorption technique is based on the absorption of radiation at 253.7 nm by mercury vapor. It relies on the volatility of elemental mercury at room temperature. During preparation, organic mercurials are oxidized and elemental mercury is ionized to Hg^{3+}. During instrumental analysis, mercuric ions are reduced to elemental mercury by the addition of stannous chloride. Elemental mercury is then aerated from solution and passes through a cell positioned in the path of a mercury spectrophotometer, where absorbance (peak height) is measured as a function of mercury concentration and recorded by the associated computer. The mercury vapor is then swept out of the instrument into an exhaust hood, where it is evacuated from the laboratory.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

3.0 INTERFERENCES

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate and potassium persulfate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Sea waters, brines, and industrial effluents high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

4.0 APPARATUS AND MATERIALS

4.1 40 mL VOA vials, for use as digestion vessels.

4.2 250 mL Pyrex media bottles with plastic screw caps, for use in digesting calibration standards.

4.3 Water bath capable of maintaining a constant temperature of 95^{o} C.

4.4 Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents

4.6 Battery powered Traceable Pocket-Size Thermometer from Fisher Scientific, NIST-traceable, covering the range from -50^{o} to 750^{o} C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.

4.7 Disposable graduated polystyrene sample cups, 200 mL capacity

4.8 CETAC M-6100 automated mercury analyzer and associated peripherals and parts

4.9 Disposable graduated dose cups, 30 mL capacity

Refer to Katahdin SOP CA-629, current revision, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer" for additional required materials.

5.0 REAGENTS

5.1 Laboratory grade reagent water – mercury-free water meeting the specifications of ASTM Type II water

5.2 Concentrated sulfuric acid, trace metals grade

5.3 Concentrated nitric acid, trace metals grade

5.4 Concentrated hydrochloric acid, trace metal grade

5.5 Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.6 Potassium persulfate solution, 5% w/v: Dissolve 50g of potassium permanganate in 1L laboratory grade reagent water. The source reagent should be labeled as suitable for use in mercury determination.

5.7 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory grade reagent water and dilute to a final volume of 1 L.

5.8 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory grade reagent water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.

5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard A must be prepared fresh monthly ,and disposed of appropriately after use. (Note: the concentrations of all stock standards must be certified by the vendors as traceable to NIST reference materials).

5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. The source of the stock standard used to prepare Intermediate Mercury Standard B must be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared fresh monthly, and disposed of appropriately after use.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Aqueous samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Aqueous (total)	P, G	250 mL	HNO_3 to pH < 2	28 days
Aqueous (dissolved)	P, G	250 mL	HNO_3 to pH < 2	28 days

[1] P = polyethylene or G = glass

7.0 PROCEDURES

BOTTLE PREPARATION

7.1 Mercury digestions are performed in two different types of vessels. Calibration standards, the Initial Calibration Verification (ICV) standard, and the Initial/Continuing Calibration Blank (ICB/CCB) are prepared in 250 mL Pyrex media bottles. Large bottles are used to provide sufficient volumes of these standards to allow for multiple reanalyses when required. Field samples, Method Blanks, and Laboratory Control Samples are digested in 40 mL VOA vials. These smaller vials provide enough digestate to allow one or two reanalyses when required, but reduce the amounts of samples consumed and waste generated.

VOA vials are reused if the samples they have contained have no measurable mercury above the PQL. After the previous contents of the vials have been discarded, these vials are segregated according to whether the measured mercury concentrations of the previous contents were above the PQL (contaminated vials) or below the PQL (uncontaminated vials). Labels are removed from the vials by wiping

with a paper towel saturated with toluene. Uncontaminated vials are rinsed with laboratory grade reagent water. Contaminated vials are discarded.

The Pyrex media bottles in which standards are prepared are emptied, rinsed, and reused. Each of these bottles is permanently marked with the concentration of the standard it contains.

PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

7.2 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS Metals database and print out a copy of the sample prep bench sheet. All necessary details of sample preparation (standards preparation information, digestion times, initial and final volumes, pertinent observations, etc.) must be recorded on this spreadsheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.

7.3 Using an industrial marker with super permanent ink, label clean VOA vials with the appropriate sample numbers and standard identifications for each sample and standard to be digested.

7.4 Use a bottle-top dispenser to add 100 mL of laboratory grade reagent water to a standard digestion bottle (250 mL media bottles). Using a calibrated adjustable pipette, prepare the high calibration standard by adding 1000 uL of Intermediate Mercury Standard A to an appropriately labeled media bottle containing 100 mL of laboratory grade reagent water. The mercury concentration of this calibration standard is 10.0 ug/L. Calibration levels 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L are made by diluting the digested 10.0 ug/L standard into calibration blank solution. See below for amounts. The 0.2 ug/L and 5 ug/L standards are analyzed after calibration as the PQL standard and the CCV (refer to Section 8.0), respectively, as well as being used in the creation of the calibration curve.

Calibration Level	Amount added	Amount calibration blank solution
0.2 ug/L	0.3 mL	14.7 mL
0.5 ug/L	0.5 mL	9.5 mL
1.0 ug/L	1 mL	9 mL
5.0 ug/L	5 mL	5 mL

7.5 Add 100 mL of laboratory grade reagent water to the media bottle labeled “ICV”. Using a calibrated adjustable pipette, prepare the Initial Calibration Verification standard (refer to Section 8) by adding 600 uL of Intermediate Mercury Standard B to the water in this bottle, and record the bottle number in the Mercury Preparation Logbook. The mercury concentration of the ICV is 6.0 ug/L.

7.6 Prepare an appropriate number of preparation blanks (PBW) by adding 25 mL of laboratory grade reagent water to labeled vials.

7.7 Prepare an appropriate number of laboratory control samples (LCSW) by adding 125 uL of Intermediate Mercury Standard A to labeled digestion vials containing 25 mL of laboratory grade reagent water. The mercury concentration of each LCSW is 5.0 ug/L.

7.8 Matrix spikes are prepared by adding 25 uL of Intermediate Mercury Std A to 25 mL aliquots of samples. The concentration of mercury added to each matrix spike is 1.0 ug/L.

7.9 All QC samples and blanks are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, sections 7.10 through 7.13 of this SOP. The volumes of reagents added to the standards prepared in the media bottles are four times those listed in sections 7.10 through 7.13.

SAMPLE PREPARATION AND DIGESTION

7.10 Using a graduated disposable dosecup, transfer 25 mL of sample, or an aliquot diluted to 25 mL, to a digestion vial. Add 1.25 mL of concentrated sulfuric acid and 0.625 mL of concentrated nitric acid, swirling to mix after each addition. Add 3.75 mL of potassium permanganate solution, swirl to mix, and allow to stand for at least 15 minutes. Samples that contain large amounts of organic substances may require additional 3.75 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 3.75 mL aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples require these additional aliquots of potassium permanganate solution, record the additional volume used for each sample on the mercury preparation benchsheet.

7.11 Add 2 mL of potassium persulfate solution to each sample. Cap the vials and place them in a preheated water bath. Monitor the temperature of the bath with a spirit thermometer throughout the digestion. The temperature of the water bath will fall below 95^{o} C upon addition of the digestion vials. After the temperature of the bath has risen back to 95^{o} C, continue heating the samples at 95^{o} C for two hours. Record initial and final digestion times and temperatures in the mercury prepareation benchsheet.

7.12 Remove bottles from the water bath and allow to cool to room temperature. If the purple permanganate color has failed to persist after digestion in any of the samples, add additional 3.75 mL aliquots of potassium permanganate solution as required to the samples, and record these additions in the mercury preparation benchsheet. Heat the samples that required additional permanganate in the water bath at 95^{o} C for an additional two hours. Remove the bottles from the water bath

and allow to cool to room temperature. If the purple color fails to persist after the second heating step, consult the Department Manager for advice on how to proceed.

7.13 Add 1.5 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion vial and swirl to mix. This will reduce the excess permanganate, and the sample will change from purple to colorless. Wait at least 30 seconds before proceeding with analysis.

INSTRUMENTAL ANALYSIS

7.14 Digested mercury samples are analyzed using the CETAC M-6100 Automated Mercury Analyzer. Analysis is automated and is controlled by the QuickTrace Mercury Analyzer software running on a dedicated PC. Detailed instructions for setting up the instrument and analyzing samples are given Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M-6100 Automated Mercury Analyzer".

METHOD OF STANDARD ADDITIONS

7.15 The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

7.15.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration C_x is calculated:

$$C_X = \frac{S_B V_S C_S}{(S_A - S_B) V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

7.15.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 3. A linear regression program may be used to obtain the intercept concentration.

7.15.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

- The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.

- The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.

- The determination must be free of spectral interference and corrected for nonspecific background interference.

DATA REDUCTION AND REPORTING

7.16 Results are obtained in concentration units (ug/L) from the instrument. Electronic instrument data files are imported into the Metals ACCESS database for data reduction. Sample preparation information (initial sample volumes and final digestate volumes) are entered directly into the Metals ACCESS database to allow calculation of final results for reporting. Results are calculated as follows:

$$\text{Mercury concentration (ug/L)} = \frac{MC \times DF \times IV}{FV}$$

where: MC = Measured mercury concentration (ug/L)
DF = Dilution factor at instrument
IV = Initial sample volume (mL)
FV = Final digestate volume (mL)

7.17 Results that exceed the calibration range of the instrument may not be reported - the sample must be appropriately diluted and reanalyzed. Results for diluted samples should be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the resulting dilution must be corrected for before reporting.

7.18 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported to the PQL and flagged with a “U” qualifier.

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 7470 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.4 through 7.8 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The Department Manager, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

In some cases the standard QC requirements listed in Table 1 may not be sufficient to meet the Data Quality Objectives of the specific project. Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.
INITIAL DEMONSTRATION OF PERFORMANCE

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate analyses of a laboratory grade reagent water spiked at 3-5 times the anticipated detection limit for each analyte, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of laboratory grade reagent water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

8.4 Instrument calibration - The instrument must be calibrated each time it is set up, and calibration standards must be digested each day that samples are digested. Calibration includes analysis of a calibration blank and five calibration standards with graduated concentrations in the appropriate range. The concentration of one of the calibration standards must be at the Practical Quantitation Level (PQL). The intermediate standards used for preparing the calibration standards are prepared at least once per month in 2% nitric acid. Because mercury may be adsorbed onto the walls of glass and plastic containers, the calibration standards must be prepared fresh daily. The correlation coefficient for the calibration curve must be at least 0.995. If the calibration curve does not pass this test, analysis must be halted, the problem corrected, and the instrument recalibrated.

8.5 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110% of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.

8.6 The Continuing Calibration Verification (CCV) solution is analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standard used for calibration at a concentration

near the mid-point of the calibration curve. Results of the CCVs must fall within 80% to 120% of the expected value. If a CCV fails, associated sample results may not be reported from the run until the problem is corrected and a passing CCV has been analyzed. Also, all samples analyzed after the last passing CCV must be reanalyzed.

8.7 A calibration blank is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If samples are being run using DoD QSM criteria, the absolute values of ICBs and CCBs must be less than the Limit of Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed. Also, all samples analyzed after the last passing CCB must be reanalyzed.

8.8 A standard with a mercury concentration that is at the Practical Quantitation Limit (PQL) is analyzed at the beginning of the run to determine calibration accuracy at the reporting limit. Result of the PQL standard should fall within 70% to 130% of the expected values. No corrective action has been established at this time.

PREPARATION BATCH QC SAMPLES

8.9 Preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.10 A laboratory control sample (LCSW), consisting of spiked reagent carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. Results for laboratory control samples must fall within 80% to 120% of the expected value, unless laboratory-generated statistical limits are available. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested.

SAMPLE MATRIX QC SAMPLES

8.11 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. Matrix spike recoveries for these samples are calculated as follows:

$$\text{Recovery (\%)} = \frac{(P - S)}{A} \times 100\%$$

where: P = Spiked sample value
S = Original sample value
A = Spike amount

The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, a matrix interference should be suspected and the associated sample result should be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$\text{RPD (\%)} = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = Spike sample result
D_2= Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

8.12 A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL or 50 x LOQ if using DoD QSM acceptance criteria), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$\text{Difference (\%)} = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

8.13 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the LOQ. The recovery of the PDS must be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and then verified one time per type of instrument.

Limits of Detection (LODs) must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verifified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The LOQs/PQLs shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO.
LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of USEPA Method 7470 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Wastes, United States Environmental Protection Agency, USEPA SW 846, Third Edition, Final Update III (9/94), Method 7470A.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Current Version.

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications.

QuickTrace M6100 Mercury Analyzer Operator Manual Version 1.0.1, CETAC Technologies.

QuickTrace Mercury Analyzer Software Manual, CETAC Technologies.

List of Tables and Figures

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA 7470	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient $\geq$ 0.995.	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within $\pm$ 10% of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within $\pm$ 30% of true value.	Correct problem and repeat calibration.
	Continuing Calibration Verification (CCV)	At beginning or run, after every 10 samples, and at end of the run	Recovery within $\pm$ 10% of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning or run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBW)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration $\geq$ PQL and < 10x the blank concentration.
	Laboratory Control Sample (LCSW)	One per digestion batch of 20 or fewer samples.	Recovery within $\pm$ 20% of true value.	Redigest all affected samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery $\pm$25% of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P)	One per digestion batch of 20 or fewer samples.	1) Recovery $\pm$ 25% of true value, if sample < 4x spike added. 2) RPD ≤20% for duplicate spikes.	Flag results
	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1) Repeat IDL study. 2) Raise PQL.
	Limit of Detection (LOD) determination	Quarterly.	LOD = 2-3X MDL	Repeat LOD Determination.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

TABLE 2

DOD QSM VERSION REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	(Refer to current revision of SOP QA-806)				
LOQ establishment and verification	(Refer to current revision of SOP QA-806)				
Initial calibration (ICAL) for mercury - minimum 5 standards and a calibration blank	Daily ICAL prior to sample analysis.	5 points plus a calibration blank, r ≥ 0.995.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analyte(s) within ± 10% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	within ± 20% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

TABLE 2

DOD QSM VERSION REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL (> RL for common lab contaminants) and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	Problem must be corrected. All samples following the last acceptable calibration blank must be reanalyzed.
LCS	One per preparatory batch.	Water: Recovery must be within + 20% of the true value Soil: Recovery must be within vendor supplied limits (varies by lot).	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available (see full explanation in Appendix G).	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	Recovery must be within + 20% of the true value.	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix (see Box D-7).	MSD: Recovery must be within + 20% of the true value. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

TABLE 2

DOD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

TABLE 3

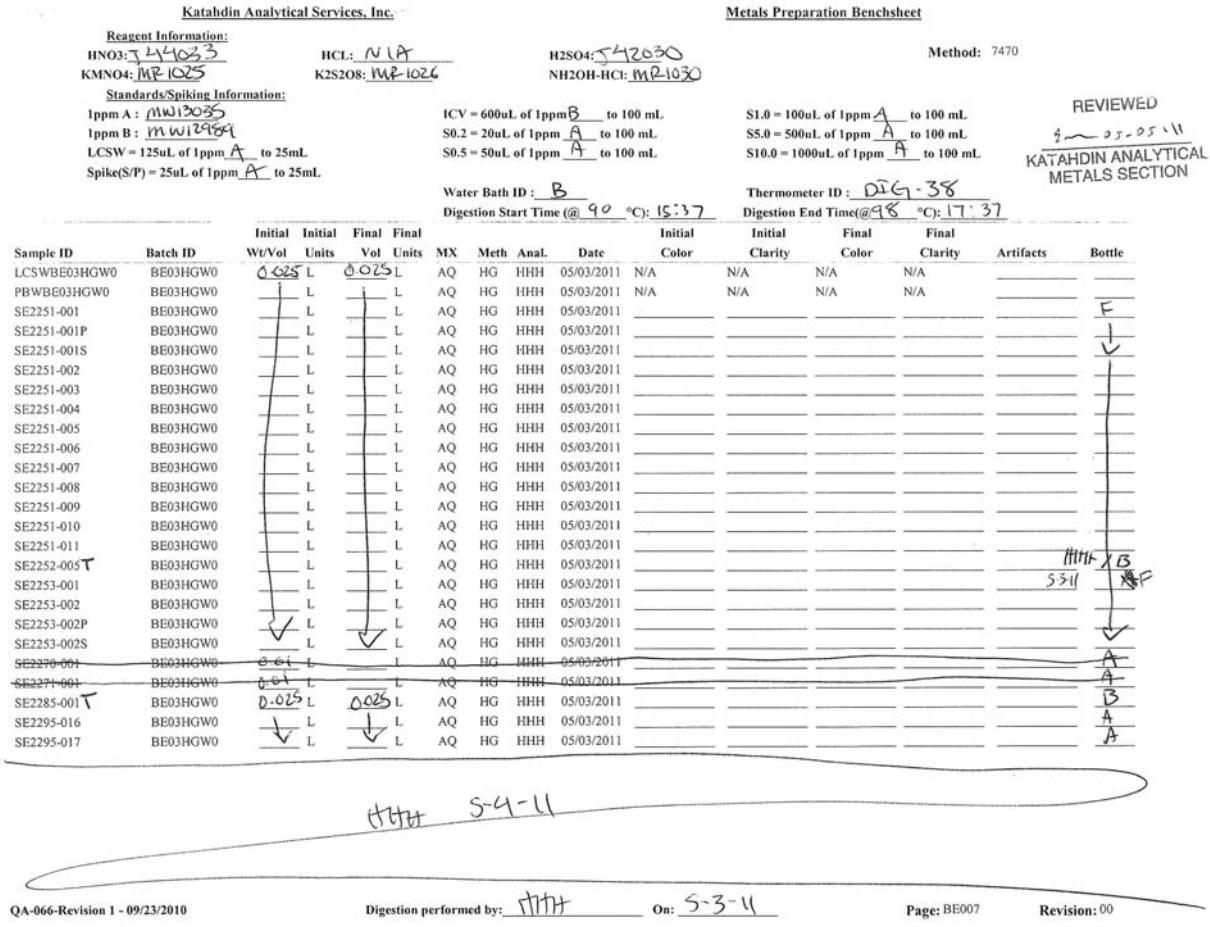
SUMMARY OF METHOD MODIFICATIONS

TOPIC	KATAHDIN SOP CA-615-07	USEPA METHOD 7470
Reagents	Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	1)Sampling and gas stream switching performed automatically by mercury analyzer. 2)Working Mercury standard prepared monthly in 2% nitric; calibration standards prepared fresh daily.	1)Sampling and gas stream switching performed manually by analyst. 2)Working Mercury standard prepared fresh daily and acidity maintained at 0.15% nitric.
QC – Calibration Verification	1) Known reference sample (ICV) analyzed daily. 2) Calibration verified after every 10 samples with CCV.	1) Known reference sample analyzed quarterly. 2) Calibration verified after every 20 samples.
QC - Calibration Blanks	Acceptance criteria employed for 245.1: ± PQL	Acceptance criteria stated in 245.1: ± MDL

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK



Katahdin Analytical Services, Inc.

Metals Preparation Benchsheet

Reagent Information:
HNO3: J44033 HCL: N/A H2SO4: J42030
KMNO4: MP-1025 K2S2O8: MP-1026 NH2OH-HCl: MP-1030

Method: 7470

Standards/Spiking Information:
1ppm A : MW13035
1ppm B : MW12989
LCSW = 125uL of 1ppm A to 25mL
Spike(S/P) = 25uL of 1ppm A to 25mL

ICV = 600uL of 1ppm B to 100 mL
S0.2 = 20uL of 1ppm A to 100 mL
S0.5 = 50uL of 1ppm A to 100 mL

S1.0 = 100uL of 1ppm A to 100 mL
S5.0 = 500uL of 1ppm A to 100 mL
S10.0 = 1000uL of 1ppm A to 100 mL

REVIEWED 5-05-11 KATAHDIN ANALYTICAL METALS SECTION

Water Bath ID : B
Digestion Start Time (@ 90 °C): 15:37
Thermometer ID : DIG-38
Digestion End Time(@ 98 °C): 17:37

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Clarity	Final Color	Final Clarity	Artifacts	Bottle
LCSWBE03HGW0	BE03HGW0	0.025	L	0.025	L	AQ	HG	HHH	05/03/2011	N/A	N/A	N/A	N/A		
PBWBE03HGW0	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011	N/A	N/A	N/A	N/A		
SE2251-001	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						F
SE2251-001P	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						↓
SE2251-001S	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						
SE2251-002	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						
SE2251-003	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						
SE2251-004	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						
SE2251-005	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						
SE2251-006	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						
SE2251-007	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						
SE2251-008	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						
SE2251-009	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						
SE2251-010	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						
SE2251-011	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						
SE2252-005T	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011					HHH 5-3-11	B
SE2253-001	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						
SE2253-002	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						
SE2253-002P	BE03HGW0	✓	L	✓	L	AQ	HG	HHH	05/03/2011						↓
SE2253-002S	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						
~~SE2270-001~~	~~BE03HGW0~~	~~0.01~~	~~L~~		~~L~~	~~AQ~~	~~HG~~	~~HHH~~	~~05/03/2011~~						~~A~~
~~SE2271-001~~	~~BE03HGW0~~	~~0.01~~	~~L~~		~~L~~	~~AQ~~	~~HG~~	~~HHH~~	~~05/03/2011~~						~~A~~
SE2285-001T	BE03HGW0	0.025	L	0.025	L	AQ	HG	HHH	05/03/2011						B
SE2295-016	BE03HGW0		L		L	AQ	HG	HHH	05/03/2011						A
SE2295-017	BE03HGW0	↓	L	↓	L	AQ	HG	HHH	05/03/2011						A

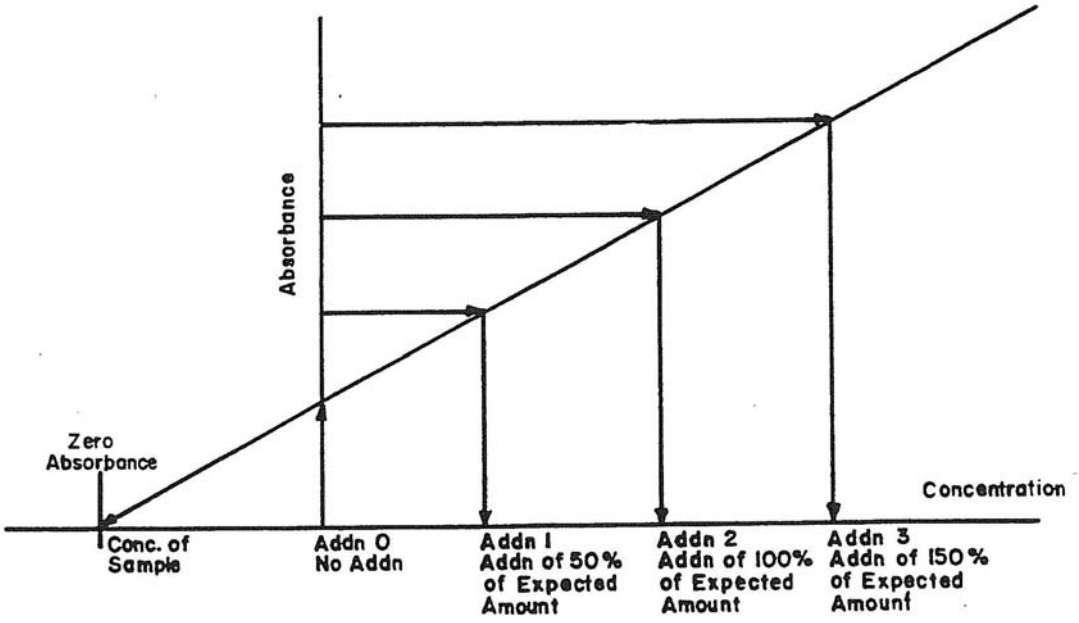
HHH 5-4-11

QA-066-Revision 1 - 09/23/2010 Digestion performed by: HHH On: 5-3-11 Page: BE007 Revision: 00

TITLE: DIGESTION AND ANALYSIS OF AQUEOUS SAMPLES FOR MERCURY BY USEPA METHOD 7470

FIGURE 2

STANDARD ADDITIONS PLOT



ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Nicholas Tillotson

Review Date: 1/22/13

SOP Number: CA-615-07

SOP Title: Digestion and analysis of aqueous samples for Mercury by US EPA method 7470

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 02/26/13

QAO Signature: Susie Dimond **Date:** 030413

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Prepared By: George Brewer Date: 12/97

Approved By:

Group Supervisor: George Brewer Date: 01/29/01

Operations Manager: John C. Busta Date: 1/29/01

QA Officer: Deborah J. Nadeau Date: 1.29.01

General Manager: Dennis F. Murphy Date: 1/29/01

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
02 7471A	Format changes, added pollution prevention, other minor changes to sections 7,8 and QA Table.	DN	1.29.01	1/29/01
03 7471A	Changed Leeman PS200 Automated Mercury Analyzer to Cetac M6100 Mercury analyzer. Revised Sect. 10 to show correct reference material. Removed fig. 2 Revised sect. 4.8, 5.7 and 8.9 to reflect current practises. minor changes through out	LAD	021605	021605
04 7471A	Sect. 5.9 and 5.10 - changed preparation of intermediate mercury standards from daily to monthly. Sect. 7.8 - removed calibration blanks (CCB/CCB). They are prepared in sect. 7.6. Added weighing of boiling chips for the prep blanks. Sect. 8.3 - Removed intermediate standards	LAD	03/08	03/08
05	Revised Sections 8 and 10, and Tables 1 and 2 to update compliance from method 7471A to method 7471B.	LAD	02/09	02/09
06	Added LOD definition. Updated sections 8, 9, 10 and Table 1 for DoD QSM version 4.1 compliance.	DN	08/09	08/09

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Revision History:

SOP Revision	Changes	Approval Initials	Approval Date	Effective Date
07	Added Table 2 with DoD QSM Version 4.1 QC Requirements	LAD	04/10	04/10
08	Sect. 4.6 - Changed thermometer type. Added LCS0 - A LCS prepped using aqueous mercury LCS spike. Updated type of marker used to label digestion bottles. Updated corrective action for failing PQL standard.	LAD	12/10	12/10
09	Sect. 7 - Changed calibration digestion from digestion of all points to digestion of high-point and dilution of rest. Changed prep from 3 ± 0.2g aliquots to 1 ± 0.6g aliquot. Added additional prep info. Added Serial dilution and PDS to sect. 8, Added MDL, LOD, LOQ info. to sect. 9. Updated and added references to Sect. 10.	LAD	04/12	04/12

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

Please acknowledge receipt of this standard operating procedure by signing and dating both of the spaces provided. Return the bottom half of this sheet to the QA Department.

I acknowledge receipt of copy ___ of document SOP CA-611-09, Titled Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471.

Recipient: _____ Date: _____

KATAHDIN ANALYTICAL SERVICES, INC.
STANDARD OPERATING PROCEDURE

I acknowledge receipt of copy ___ of document SOP CA-611-09, Titled Digestion and Analysis of Solid Samples for Mercury by USEPA Method 7471.

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Recipient: _____ Date: _____

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to describe the procedure used by Katahdin Analytical Services, Inc. personnel for the digestion and analysis solid samples for mercury using cold vapor atomic absorption spectrophotometry.

This method is applicable to the determination of mercury in soils, sediments, bottom deposits, and sludges under USEPA Method 7471 (Test Method for Evaluating Solid Wastes, USEPA SW 846, Third Edition).

1.1 Definitions

ICB - Initial Calibration Blank - An analyte-free solution consisting of acidified laboratory reagent grade water used to verify calibration accuracy.

CCB - Continuing Calibration Blank - An analyte-free solution consisting of acidified laboratory reagent grade water used to verify calibration accuracy periodically during analysis.

ICV - Initial Calibration Verification - A standard made from a source independent from the calibration standards and with analyte concentrations different from those in the CCV; used to verify the accuracy of the instrument calibration.

CCV - Continuing Calibration Verification - A midrange standard used to verify calibration accuracy periodically during analysis.

LCS - Laboratory Control Sample - A standard or solid reference material that has been brought through the sample preparation process. LCSS utilizes the standard reference material. LCSO is spiked with aqueous mercury LCS spike.

PB - Preparation Blank - Laboratory reagent grade water that has been brought through the sample preparation process.

Matrix Spike - An aliquot of a sample to which a known amount of analyte has been added before digestion.

Duplicate - A second aliquot of a sample that is prepared and analyzed in the same way as the original sample in order to determine the precision of the method.

SERIAL DILUTION - The dilution of a sample by a factor of five. When corrected by the dilution factor, the measured analyte concentrations of the diluted sample should agree with those of the original undiluted sample within specified limits. Serial dilution may reflect the influence of interferents.

IDL - Instrument Detection Limit - The lowest concentration of an analyte that can be determined with 95% confidence by the instrument.

MDL - Method Detection Limit - The minimum concentration of an analyte that can be identified, measured, and reported with 99% confidence that the analyte concentration is greater than zero.

LOD – Limit of Detection – An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix-specific and is used for DoD QSM acceptance criteria.

PQL - Practical Quantitation Limit - The lowest concentration of an analyte that is routinely reported by the laboratory; nominally three to five times the IDL.

1.2 Responsibilities

This method is restricted to use by, or under the supervision of analysts experienced in the analysis of mercury by USEPA Method 7471. Each analyst must demonstrate and document their ability to generate acceptable results with this method. Refer to Katahdin SOP QA-805, current revision, “Personnel Training & Documentation of Capability”.

It is the responsibility of all Katahdin technical personnel involved in analysis of mercury by USEPA Method 7471 to read and understand this SOP, to adhere to the procedures outlined, and to properly document their data in the appropriate lab notebook. Any deviations from the test or irregularities with the samples should also be recorded in the lab notebook and reported to the Department Manager or designated qualified data reviewer responsible for this data.

It is the responsibility of the Department Manager to ensure that members of their group follow this SOP, to ensure that their work is properly documented, and to initiate periodic review of the associated logbooks.

1.3 Safety

Many of the samples and reagents used in cold vapor atomic absorption are toxic or corrosive. Gloves, safety glasses, lab coats, and other protective clothing should be worn whenever these materials are handled. Because of the toxic nature of mercury vapor, care must be taken to avoid its inhalation. The instrument exhaust fan must be in operation whenever the mercury analyzer is in use (the fan should never be shut off).

Users of this procedure must be cognizant of inherent laboratory hazards, proper disposal procedures for contaminated materials and appropriate segregation of hazardous wastes. The toxicity or carcinogenicity of each reagent used in this

method has not been precisely defined; however, each chemical should be treated as a potential health hazard. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Everyone involved with the procedure must be familiar with the MSDSs for all the materials used in this procedure.

Each qualified analyst or technician must be familiar with Katahdin Analytical Environmental Health and Safety Manual including the Katahdin Hazardous Waste Plan and must follow appropriate procedures. These include the use of appropriate personal protective equipment (PPE) such as safety glasses, gloves and lab coats when working with chemicals or near an instrument and not taking food or drink into the laboratory. Each analyst should know the location of all safety equipment. Each analyst shall receive a safety orientation from their Department Manager, or designee, appropriate for the job functions they will perform.

1.4 Pollution Prevention/Waste Disposal

Whenever possible, laboratory personnel should use pollution prevention techniques to address there waste generation. Refer to the current revision of the Katahdin Hazardous Waste Management Plan for further details on pollution prevention techniques.

Samples, sample digestates, standards, and other reagents used in cold vapor atomic absorption may contain high concentrations of acids, mercury, and other toxic metals. They should be disposed of in a manner appropriate to the types of hazards they present. All digested mercury samples and standards and excess reagents and standards should be disposed of in the satellite waste container for corrosive wastes (labeled “Waste Stream A”) that is located in the Metals Prep lab. Further information regarding waste classification and disposal may be obtained by consulting the laboratory's Katahdin Analytical Environmental Health and Safety Manual and the Department Manager.

2.0 SUMMARY OF METHOD

The cold vapor atomic absorption technique is based on the absorption of radiation at 253.7 nm by mercury vapor. It relies on the volatility of elemental mercury at room temperature. During preparation, organic mercurials are oxidized and elemental mercury is ionized to Hg^{3+}. During instrumental analysis, mercuric ions are reduced to elemental mercury by the addition of stannous chloride. Elemental mercury is then aerated from solution and passes through a cell positioned in the path of a mercury spectrophotometer, where absorbance (peak height) is measured as a function of mercury concentration and recorded by the associated computer. The mercury vapor is then swept out of the instrument into an exhaust hood, where it is evacuated from the laboratory.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

3.0 INTERFERENCES

In addition to inorganic forms of mercury, organic mercurials may be present in environmental samples. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. The presence of undigested organo-mercurials in samples will result in a low bias for analytical results. Certain volatile organic materials will also non-specifically absorb radiation at the 253.7 nm analytical wavelength. The presence of such compounds may result in a high bias for analytical results. For these reasons, complete digestion using potassium permanganate is required for all environmental samples. Complete digestion is indicated by the persistence of the purple permanganate color (indicating the presence of excess permanganate) following digestion.

Samples that are high in chlorides may require additional permanganate to maintain a persistent purple color following digestion. During the oxidation step, chlorides are converted to free chlorine, which will absorb radiation at the 253.7 nm analytical wavelength. Any free chlorine thus generated will be present in the headspace of the digestion vessel following digestion. Because samples are poured into autosampler tubes prior to analysis by the mercury analyzer, any free chlorine present in the headspace of the digestion vessels is not sampled by the instrument and the analysis is free of chlorine interference.

4.0 APPARATUS AND MATERIALS

4.1 250 mL Pyrex media bottles with plastic screw caps, for use as digestion vessels.

4.2 Water bath capable of maintaining a constant temperature of 95^{o} C.

4.3 Analytical balance capable of weighing to 0.01 g.

4.4 Adjustable volume automatic pipettes - 2 to 20 uL, 10 to 100 uL, 100 to 1000 uL. Calibrated Eppendorf Reference pipets and Finn digital pipets are appropriate.

4.5 Repipetters (adjustable repeating pipetters with reservoirs) for dispensing concentrated nitric acid, concentrated sulfuric acid, and other reagents.

4.6 Battery powered Traceable Pocket-Size Thermometer from Fisher Scientific, NIST-traceable, covering the range from -50^{o} to 750^{o} C, for monitoring the temperature of the water bath. Mercury-filled thermometers are not acceptable for use in the metals laboratory, due to the possibility of breakage and consequent contamination.

4.7 Disposable graduated polystyrene sample cups, 200 mL capacity.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

4.8 CETAC M6100 Mercury Analyzer and associated peripherals and parts.

Refer to Katahdin SOP CA-629, current revision, "Operation and Maintenance of the CETAC M6100 Mercury Analyzer" for additional required materials.

5.0 REAGENTS

5.1 Laboratory reagent grade water – mercury-free water.

5.2 Concentrated nitric acid (HNO_3), trace metal grade

5.3 Concentrated hydrochloric acid (HCl), trace metal grade

5.4 Aqua regia: Prepare an appropriate amount immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3 in a heat-proof beaker or flask. Preparation of aqua regia must be performed in a fume hood.

5.5 Potassium permanganate solution, 5% w/v: Dissolve 50 g of potassium permanganate in 1 L laboratory reagent grade water. The source reagent should be labeled as suitable for use in mercury determination.

5.6 Sodium chloride – hydroxylamine hydrochloride solution: Dissolve 120 g sodium chloride and 120 g hydroxylamine hydrochloride in laboratory reagent grade water and dilute to a final volume of 1 L.

5.7 Stannous chloride solution: Add 70 mL concentrated hydrochloric acid to 500 mL of laboratory reagent grade water. Add 100 g stannous chloride and bring to a final volume of 1 L. Mix to dissolve. Reagent should be labeled as suitable for use in mercury determination.

5.8 Mercury Stock Standards: Two 10.0 mg/L mercury stock standards, obtained from separate sources, are required. The mercury concentrations of these standards must be certified by the manufacturers as traceable to NIST reference standards.

5.9 Intermediate Mercury Standard A: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. This intermediate standard is used to prepare calibration standards, matrix spikes, CCVs, and laboratory control samples (refer to Section 8). The identity of the stock standard currently used to prepare this intermediate standard and instructions for its dilution may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard A must be prepared monthly, and disposed of appropriately after use. (Note: the concentrations of all

stock standards must be certified by the vendors as traceable to NIST reference materials).

5.10 Intermediate Mercury Standard B: Appropriately dilute a mercury stock standard to obtain a solution containing 1000 ug of mercury per liter in 2% nitric acid. The source of the stock standard used to prepare Intermediate Mercury Standard B must be distinct from that used to prepare Intermediate Mercury Standard A (i.e. obtained from a separate vendor). Intermediate Mercury Standard B is used to prepare the ICV (refer to Section 8.0). The identity of the stock standard currently used to prepare this intermediate standard and instructions for its dilution may be obtained by consulting the Standards Preparation Logbook maintained in the Section. Intermediate Mercury Standard B must be prepared monthly, and disposed of appropriately after use.

5.11 Solid Reference Material: A soil with a known or empirically-established mercury concentration for use in preparing the laboratory control sample for soils. Solid reference materials should be purchased with certificates listing reference values and quality control acceptance limits. See Figure 3 for an example certificate of analysis for a solid reference material.

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

Soil samples to be analyzed for mercury should be collected and preserved as described in the following table.

Matrix	Container[1]	Collection Volume/ Weight	Preservation/ Treatment	Holding Time
Solid	P, G	40 g	Cool to 4°C ± 2°	28 days

[1] P = polyethylene, G = glass

7.0 PROCEDURES

BOTTLE PREPARATION

7.1 Mercury digestion bottles are reused, and must be cleaned between uses. After the previous contents of the bottles have been discarded, bottles are segregated according to whether the measured mercury concentrations of the previous contents were above the PQL (contaminated bottles) or below the PQL (uncontaminated bottles). Labels are removed from the bottles by wiping with a paper towel saturated with toluene. Both contaminated and uncontaminated bottles are then cleaned with Liquinox and water, if necessary, to remove visible grime, and rinsed thoroughly with tap water.

7.2 Uncontaminated bottles are then triple-rinsed with laboratory reagent grade water, and are ready for reuse.

7.3 Contaminated bottles are placed in a bath containing 10% HCl for at least 12 hours. After acid-leaching, these bottles are triple rinsed with laboratory reagent grade water, and are then ready for reuse.

PREPARATION OF STANDARDS, QC SAMPLES, AND BLANKS

7.4 Prior to performing the digestion, make a list of the samples that are to be digested. Enter digestion information (Katahdin Sample Numbers, Bottle IDs, QC Batch ID, preparation date, analyst initials, etc.) into the ACCESS computer database and print out a copy of the benchsheet. All necessary details of sample preparation (standards preparation information, digestion times, digestion temps, initial weights and final volumes, pertinent observations, etc.) must be recorded on this benchsheet, which will be bound in the Mercury Preparation Logbook. Refer to Figure 1 for an example page from the Mercury Preparation Logbook.

7.5 Using an industrial marker with super permanent ink, label clean digestion bottles with the appropriate sample numbers and standard identifications for each sample and standard to be digested.

7.6 Use a bottle-top dispenser to add 100 mL of laboratory grade reagent water to a standard digestion bottle (250 mL media bottles). Using a calibrated adjustable pipette, prepare the high calibration standard by adding 1000 uL of Intermediate Mercury Standard A to an appropriately labeled media bottle containing 100 mL of laboratory grade reagent water. The mercury concentration of this calibration standard is 10.0 ug/L. Calibration levels 0.2 ug/L, 0.5 ug/L, 1.0 ug/L, 5.0 ug/L are made by diluting the digested 10.0 ug/L standard into calibration blank solution. See below for amounts. The 0.2 ug/L and 5.0 ug/L standards are analyzed after calibration as the PQL standard and the CCV (refer to Section 8.0), respectively, as well as being used in the creation of the calibration curve.

Calibration Level	Amount added	Amount Calibration Blank Solution
0.2 ug/L	0.3 mL	14.7 mL
0.5 ug/L	0.3 mL	9.5 mL
1.0 ug/L	1 mL	9 mL
5.0 ug/L	5 mL	5 mL

7.7 Using a calibrated adjustable pipette, prepare the initial calibration verification (ICV) standard (refer to Section 8) by adding 600 uL of Intermediate Mercury Standard B to an appropriately labeled digestion bottle. The mercury concentration of the ICV will be 6.0 ug/L.

7.8 Prepare an appropriate number of preparation blanks (PBS) by adding 1.0 g of Teflon boiling chips to labeled digestion bottles.

7.9 Prepare an appropriate number of laboratory control samples (LCSS or LCSO) by weighing appropriate masses of solid reference material or by adding 500 uL of Intermediate Mercury Standard A respectively into labeled digestion bottles. The mercury concentration of the LCSS will depend on the solid reference material used, and the mass of each aliquot. Refer to Figure 3 for an example certificate of analysis for a solid reference material. The mercury concentration of the LCSO will be 5.0 ug/L.

7.10 Matrix spikes are prepared by adding 100 uL of Intermediate Mercury Std A to each matrix spike sample. The amount of mercury added to each matrix spike increases the final digestate concentration by 1.0 ug/L.

7.11 All calibration standards, QC samples, and blanks are digested in the same manner as client samples. Refer to Sample Preparation and Digestion, Steps 7.12 through 7.16 of this SOP.

SAMPLE PREPARATION AND DIGESTION

7.12 Do not decant any water on the sediment sample. Mix sample with a wooden spatula to ensure homogeneity of the sample. Please refer to the current revision of Katahdin Analytical Services SOP CA-108, “Basic Laboratory Technique“, for more detailed guidance on sub-sampling to ensure reproducibility.

Weigh an approximate 0.6 g portion of untreated, homogenized sample from the sample container and place in the bottom of a labeled digestion bottle.

7.13 Add 5 mL of laboratory reagent grade water and 5 mL of aqua regia to each sample, standard, and QC sample. Place bottles in a water bath located in a fume hood and heat for 2 minutes at 95^{o} C. Remove the bottles from the water bath and allow them to cool in a fume hood.

7.14 Add 50 mL of laboratory reagent grade water and 15 mL of potassium permanganate solution to each digestion bottle, swirl to mix, and allow to stand for at least 15 minutes. Samples that contain large amounts of oxidizable organic matter may require additional 15 mL aliquots of potassium permanganate solution. This is indicated by the failure of the purple permanganate color to persist for the entire 15 minute waiting period. Add additional 15 mL aliquots to samples as necessary until the purple color persists for 15 minutes. If any of the samples requires these additional aliquots of permanganate, note that fact on the mercury preparation benchsheet and accordingly adjust the final volumes recorded on the benchsheet for those samples.

When a persistent purple color has been obtained for all samples, place the digestion bottles in the water bath and heat for 30 minutes at 95^{o} C. Record initial and final time and temperatures on the mercury preparation benchsheet.

7.15 Remove the bottles from water bath and allow them to cool in a fume hood. If any of the samples have become colorless during heating, add additional 15 mL aliquots of potassium permanganate solution as necessary to obtain a persistent purple color and heat for an additional 30 minutes at 95^{o} C. Record any information regarding additional permanganate aliquots on the mercury preparation benchsheet and accordingly adjust the final volumes recorded on the benchsheet for the samples affected.

7.16 Add 6 mL of sodium chloride – hydroxylamine hydrochloride solution to each digestion bottle and swirl to mix. Perform this addition in a fume hood, as chlorine gas may be evolved. This will reduce the excess permanganate, and the sample will change from purple to colorless. Add 50 mL of laboratory reagent grade water to each bottle. Wait at least 30 seconds before proceeding with analysis.

INSTRUMENTAL ANALYSIS

7.17 Digested mercury samples are analyzed using the CETAC M6100 Mercury Analyzer. Analysis is automated and is controlled by the QuickTrace software running on a dedicated PC. Detailed instructions for setting up the instrument and running samples are given Katahdin SOP CA-629, “Operation and Maintenance of the CETAC M6100 Mercury Analyzer”. The following information specifically pertains to analysis of digested samples in accordance with USEPA Method 7471, and should be used in conjunction with the instructions given in Katahdin SOP CA-629.

7.18 Instrument operating conditions and quality control acceptance limits are specified in the instrument software in “templates”. The template that is used to analyze digested samples in accordance with USEPA Method 7471 is named “SW846-7470-7471”.

7.19 Prior to analysis, digested samples, standards, and QC samples are decanted into autosampler tubes which are placed in racks on the instrument’s autosampler. The “standards” autosampler rack has 10 positions for 25 x 100 mm autosampler tubes (50 mL capacity). Tubes containing the calibration standards, the ICV, the CCV, the ICB/CCB, and the PQL standard are placed in the appropriately labeled positions in this autosampler rack.

7.20 Client samples, batch QC samples (preparation blanks and laboratory control samples), and matrix QC samples (duplicates and matrix spikes) are decanted into 17 x 100 mm autosampler tubes (15 mL capacity), which are placed in the one of

the "samples" autosampler racks. The "samples" autosampler racks have 60 positions for 17 x 100 mm autosampler tubes. Instructions for filling the "samples" autosampler racks, including recording the rack position of each sample, are contained in Katahdin SOP CA-629, "Operation and Maintenance of the CETAC M6100 Mercury Analyzer".

METHOD OF STANDARD ADDITIONS

7.21 The standard addition technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constituent that enhances or depresses the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences that cause a baseline shift. The method of standard additions shall be used for analysis of all EP extracts, on all analyses submitted as part of a delisting petition, and whenever a new sample matrix is being analyzed.

7.21.1 The simplest version of this technique is the single-addition method, in which two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a known volume V_S of a standard analyte solution of concentration C_S. To the second aliquot (labeled B) is added the same volume V_S of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration C_x is calculated:

$$C_X = \frac{S_B V_S C_S}{(S_A - S_B) V_X}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and C_s should be chosen so that S_A is roughly twice S_B on the average, avoiding excess dilution of the sample. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure.

7.21.2 Improved results can be obtained by employing a series of standard additions. To equal volumes of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume. For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance. The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is

extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. An example of a plot so obtained is shown in Figure 2. A linear regression program may be used to obtain the intercept concentration.

7.21.3 For the results of this MSA technique to be valid, the following limitations must be taken into consideration:

- The apparent concentrations from the calibration curve must be linear over the concentration range of concern. For the best results, the slope of the MSA plot should be nearly the same as the slope of the standard curve. If the slope is significantly different (greater than 20%), caution should be exercised.

- The effect of the interference should not vary as the ratio of analyte concentration to sample matrix changes, and the standard addition should respond in a similar manner as the analyte.
- The determination must be free of spectral interference and corrected for nonspecific background interference.

DATA REDUCTION AND REPORTING

7.22 Results are obtained in units of ug/L in the digestate. Results that exceed the calibration range of the instrument may not be reported - the sample must be appropriately diluted and reanalyzed. Results for diluted samples must be multiplied by the dilution factor prior to reporting. If additional aliquots of potassium permanganate were added during digestion, the change in digestate final volume must be taken into account in calculating the final result. Mercury results for solid samples are reported in units of ug/g, calculated on a dry weight basis. Calculation of mercury results for solid samples is performed automatically by the Metals reporting database, as follows:

$$\text{Mercury Concentration in Solid (mg/kg dry wt.)} = \frac{(C) \times (DF) \times (FV) \times 100}{(W) \times (TS)}$$

where C = Measured digestate concentration (ug/L)
DF = Instrument dilution factor
FV = Digestate final volume (L)
W = Digested wet sample weight (g)
TS = Total Solids (%)

7.23 Results are reported down to the laboratory's practical quantitation level (PQL), unless otherwise requested. Results below the PQL should be reported as "<PQL".

8.0 QUALITY CONTROL AND ACCEPTANCE CRITERIA

USEPA Method 7471 requires the laboratory to perform specific quality control checks to assess laboratory performance and data quality. Minimum frequencies, acceptance criteria, and corrective actions for these control checks are tabulated in Table 1 and are described below. Preparation instructions and the resulting mercury concentrations for calibration standards, QC standards, and matrix spikes are detailed in Sections 7.6 through 7.10 of this SOP. Table 1 criteria are intended to be guidelines for analysts. The table does not cover all possible situations. If any of the QC requirements are outside the recovery ranges listed in Table 1, all associated samples must be evaluated against all the QC. In some cases data may be reported, but may be reanalyzed in other cases. Making new reagents and standards may be necessary if the standardization is suspect. The corrective actions listed in Table 1 may rely on analyst experience to make sound scientific judgments. These decisions are based on holding time considerations and client and project specific Data Quality Objectives. The supervisor, Operations Manager, General Manager and/or Quality Assurance Officer may be consulted to evaluate data. Some samples may not be able to be reanalyzed within hold time. In these cases “qualified” data with narration may be advisable after consultation with the client.

Much of the work performed at the lab is analyzed in accordance with specific QC requirements spelled out in a project specific Quality Assurance Project Plan (QAPP) or in a program specific Quality Systems Manual (QSM). The reporting limits, acceptance criteria and/or corrective actions may be different than those specified in this SOP. In these cases the appropriate information will be communicated to the Department Manager and/or senior chemists before initiation of the analyses so that specific product codes can be produced for the project. In addition, the work order notes for each project will describe the specific QAPP or QSM to be followed.

INITIAL DEMONSTRATION OF PERFORMANCE

8.1 Instrument detection limits (IDL) are determined quarterly for each analyte analyzed on each instrument by each method. This determination requires seven replicate analyses of laboratory reagent grade water spiked, performed on three non-consecutive days. The standard deviation of the 21 analyses is multiplied by three to obtain the IDL. For more information on performing IDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.2 Method detection limits (MDL) are determined annually for each analyte analyzed on each instrument. This determination requires at least seven replicate digestions and analyses of laboratory reagent grade water spiked at 3-5 times the anticipated MDL for each analyte. MDLs differ from IDLs in that the replicates are digested

prior to analysis, and they may be analyzed on a single day. The standard deviation of the 7 (or more) replicate analyses is multiplied by the Student's t-value to obtain the MDL. For more information on performing MDL determinations, refer to the current revision of Katahdin SOP QA-806.

8.3 Limits of Detection (LOD) are used when evaluating data using DoD QSM. The LOD is established by spiking a quality system matrix at 2-3 times the detection limit for a single analyte standard and 1-4 times the detection limit for a multi-analyte standard. The LOD must be verified quarterly. For more information on performing LOD determinations, refer to the current revision of Katahdin SOP QA-806.

ANALYTICAL RUN QC

8.4 Instrument calibration - The instrument must be calibrated each time it is set up, and calibration standards must be digested each day that samples are digested. Calibration includes analysis of a calibration blank and five calibration standards with graduated concentrations in the appropriate range. The concentration of one of the calibration standards must be at the Practical Quantitation Level (PQL). The correlation coefficient for the calibration curve must be at least 0.995. If the calibration curve does not pass this test, analysis must be halted, the problem corrected, and the instrument recalibrated.

8.5 An Initial Calibration Verification (ICV) solution is analyzed after the initial calibration to check calibration accuracy. The ICV solution is prepared from a standard source different than that of the calibration standard and at a concentration within the working range of the instrument. The result of the ICV must fall within 90% to 110% of the expected value. If the ICV fails, results may not be reported from the run until the problem is corrected and a passing ICV has been analyzed.

8.6 The Continuing Calibration Verification (CCV) solution is analyzed after the initial calibration, after every ten samples, and at the end of the analytical run. The CCV solution is prepared using the same standard used for calibration at a concentration near the mid-point of the calibration curve. Results of the CCVs must fall within 90% to 110% of the expected value. If a CCV fails, associated sample results may not be reported from the run until the problem is corrected and a passing CCV has been analyzed. Also, all samples analyzed after the last passing CCV must be reanalyzed. For DoD QSM acceptance criteria, samples that are below the reporting limit may be reported if the CCV reads greater than 120%.

8.7 A calibration blank is analyzed after each ICV and CCV. A calibration blank that is analyzed after the ICV is called an Initial Calibration Blank (ICB). A calibration blank that is analyzed after a CCV is called a Continuing Calibration Blank (CCB). The absolute values of results of ICBs and CCBs must be less than the Practical Quantitation Level (PQL) for each element. If samples are being run using DoD QSM criteria, the absolute values of ICBs and CCBs must be less than the Limit of

Detection (LOD). If an ICB or a CCB fails, results for the failing elements may not be reported from the run until the problem is corrected and a passing ICB or CCB has been analyzed. Also, all samples analyzed after the last passing CCB must be reanalyzed.

8.8 A standard with a mercury concentration that is at the Practical Quantitation Limit (PQL) is analyzed at the beginning of the run to determine calibration accuracy at the reporting limit. Result of the PQL standard should fall within 70% to 130% of the expected values. If the PQL fails, results may not be reported from the run until the problem is corrected and a passing PQL has been analyzed.

PREPARATION BATCH QC SAMPLES

8.9 Preparation blank (PBW or PBS), consisting of reagent water carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. The results of preparation blanks must be less than the Practical Quantitation Level (PQL) for each element. For DoD QSM acceptance criteria the results must be less than ½ the PQL except for common contaminants which must be less than the PQL. If a preparation blank fails, results for the failing elements may not be reported from the digestion batch, and all associated samples must be redigested, with the following exception. If the result for a preparation blank is greater than the PQL (greater than ½ PQL for DoD), associated sample results that are less than the PQL (less than ½ PQL for DoD) or greater than or equal to ten times the measured preparation blank concentration may be reported.

8.10 A laboratory control sample (LCSS or LCSO), consisting of solid reference material or 500 uL of Intermediate Standard A carried through the same process as associated samples, is prepared with each digestion batch of twenty or fewer samples. If a laboratory control sample fails, results may not be reported from the digestion batch, and all associated samples must be redigested. The laboratory uses a reference value and statistical acceptance limits for laboratory control samples that are supplied by the vendor of the solid reference material. The results of the LCSO must fall with in 80% - 120% of its true value which is 5.0 ug/L. If samples are being prepared using DoD QSM acceptance criteria, the results of the LCSS or LCSO must be within 80% - 120%.

SAMPLE MATRIX QC SAMPLES

8.11 Matrix spiked duplicate samples are prepared at a minimum frequency of one per digestion batch. Matrix spike recoveries for these samples are calculated as follows:

$$\text{Recovery (\%)} = \frac{(P - S)}{A} \times 100\%$$

where: P = Spiked sample value
S = Original sample value
A = Spike amount

The recovery for each element in a spiked sample or spiked duplicate sample must fall within 75% to 125% of the actual value if the result for the unspiked sample is less than four times the amount of spike added. If one or both spike recoveries fail, a matrix interference should be suspected and the associated sample result should be flagged on the report of analysis. If DoD QSM acceptance criteria are being used, recoveries must be the same as stated for laboratory control samples.

The relative percent difference between matrix spiked duplicate sample results is calculated as follows:

$$RPD\ (\%) = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where: D_1 = Spike sample result
D_2= Spike duplicate sample result

A control limit of 20% RPD is applied to matrix spike duplicate analysis. If the matrix spike duplicate analysis fails, the associated sample result should be flagged on the report of analysis.

8.12 Serial Dilution - A serial dilution is analyzed to check for chemical or physical interferences. If the analyte concentration of a sample is sufficiently high (minimally, 50 x IDL or 50 x LOQ if using DoD QSM acceptance criteria), the measured concentration of a serial dilution (1:5 dilution) of the sample should agree within 90% to 110% of the original determination. The percent difference between the original sample and the serial dilution should be calculated as follows:

$$Difference\ (\%) = \frac{|L-S|}{S} * 100\%$$

where: L = Serial dilution result (corrected for dilution)
S = Original sample result

If the serial dilution analysis fails, a matrix interference should be suspected. The associated sample result should be flagged on the report of analysis or the sample should be reanalyzed at dilution to eliminate the interference.

8.13 Post-digestion Spike (PDS) additions must be performed for DoD QSM samples if the serial dilution is not within acceptance criteria or if the analyte concentrations in all samples are less than 50x the LOD. The spike addition should produce a concentration that is between 10 and 100x the LOQ. The recovery of the PDS must

be within 75-125%. If the PDS fails, all samples must be run by method of standard additions or appropriately flagged.

9.0 METHOD PERFORMANCE

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDLs shall be determined and verified one time per type of instrument unless otherwise required by the method.

A Limit of Detection (LOD) is an estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte and matrix specific and may be laboratory-dependent. LODs must be determined for all parameters for which the laboratory is accredited under the DoD Environmental Laboratory Accreditation Program. LOD's must be verified for every preparation and analytical method combination and on every applicable instrument on a quarterly basis.

The Limit of Detection (LOQ) is the minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. The LOQ shall be set at the lowest point in the calibration curve for all analyses utilizing an initial calibration. LOQ's must be verified quarterly for every preparation and analytical method combination and on every applicable instrument on a quarterly basis for all parameters included in the DoD Scope of Accreditation. The LOQ must be verified at least once annually if the analysis is not included in the DoD Scope of Accreditation.

MDLs are filed with the Organic Department Manager and then with the QAO. LOD and LOQ verifications are filed with the QAO

Refer to the current revision of Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, for procedures on determining the MDL.

Refer to the current revision of Method 7471 for other method performance parameters and requirements.

10.0 APPLICABLE DOCUMENTS/REFERENCES

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA SW846, 3rd Edition, Final Updates I, II, IIA, IIB, III, IIIA, IIIB and IV, February 2007, Method 7471B.

Department of Defense Quality Systems Manual for Environmental Laboratories (DOD QSM), Current Version.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

The National Environmental Laboratory Accreditation Conference (NELAC) Standards, June 2003.

The NELAC Institute, Laboratory Accreditation Standards, Volume 1, Management and Technical Requirements for Laboratories Performing Environmental Analysis, 10/06/2010.

Katahdin SOP CA-101, Equipment Maintenance and Troubleshooting, current revision.

Katahdin SOP QA-806, Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications, current revision.

QuickTrace M6100 Mercury Analyzer Operator Manual Version 1.0.1, CETAC Technologies

QuickTrace Mercury Analyzer Software Manual, CETAC Technologies.

List of Tables and Figures

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 1

QC REQUIREMENTS

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA Method 7471B	Initial Calibration, 5 points plus a calibration blank.	Daily prior to sample analysis.	Correlation coefficient ≥ 0.995.	Correct problem and repeat calibration.
	Initial Calibration Verification (ICV), prepared from a second source.	Before beginning a sample run.	Recovery within ± 10% of true value.	Correct problem and repeat calibration.
	Initial Calibration Blank (ICB)	Before beginning a sample run.	Less than PQL.	Correct problem and repeat calibration.
	Practical Quantitation Level Standard (PQL)	Before beginning a sample run.	Recovery within ± 30% of true value.	Correct problem and repeat calibration.
	Continuing Calibration Verification (CCV)	At beginning or run, after every 10 samples, and at end of the run	Recovery within ± 10% of true value	Repeat calibration and reanalyze all samples analyzed since the last successful CCV.
	Continuing Calibration Blank (CCB)	At beginning or run, after every 10 samples, and at end of the run	Less than PQL.	Repeat calibration and reanalyze all samples analyzed since the last successful CCB.
	Preparation Blank (PBS)	One per digestion batch of 20 or fewer samples.	Less than PQL.	1) Investigate source of contamination. 2) Redigest and reanalyze all associated samples if sample concentration ≥ PQL and < 10x the blank concentration.
	Laboratory Control Sample (LCSS or LCSO)	One per digestion batch of 20 or fewer samples.	LCSS: Recovery within vendor-supplied acceptance limits. LCSO: Recovery within ± 20% of true value.	Redigest all affected samples.
	Matrix Spike Sample (S)	One per digestion batch of 20 or fewer samples.	Recovery ± 25% of true value, if sample > 4x spike value.	Flag results.
	Matrix Spike Duplicate Sample (P) or sample duplicate (D)	One per digestion batch of 20 or fewer samples.	1)Recovery ± 25% of true value, if sample < 4x spike added. 2) RPD ≤20% for duplicate spikes or duplicate samples.	Flag results
	Post-Digestion Matrix Spike Sample (PDS)	When matrix spike or MSD fail	Recovery ±20% of true value	Analyze serial dilution of sample
	Serial Dilution Test (L)	One per digestion batch or when PDS fails	1:5 dilution of sample must agree within 10% with undiluted result	If MS, MSD, PDS, and serial dilution fail, quantitate sample by method of standard additions

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 1

QC REQUIREMENTS (CONTINUED)

Parameter/ Method	QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mercury/ USEPA Method 7471B	Instrument Detection Limit (IDL) Study	Quarterly.	IDL < PQL	1)Repeat IDL study. 2)Raise PQL.
	Method Detection Limit (MDL) Study	Refer to KAS SOP QA-806, "Method Detection Limit, Instrument Detection Limit and Reporting Limit Studies and Verifications", current revision.		
	Limit of Detection (LOD) determination	Quarterly.	LOD = 2-3X MDL	Repeat LOD Determination.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria.	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	(Refer to current revision of SOP QA-806)				
LOQ establishment and verification	(Refer to current revision of SOP QA-806)				
Initial calibration (ICAL) for Mercury: minimum 5 standards and a calibration blank	Daily ICAL prior to sample analysis.	If more than one calibration standard is used, $r \geq 0.995$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed.
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Value of second source for all analyte(s) within ± 10% of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until calibration has been verified.
Continuing calibration verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	CVAA: within ± 20% of true value.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL and > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. Contact Client if samples cannot be reprepped within hold time. For negative blanks, absolute value < LOD.	Correct the problem. Report sample results that are <LOD or >10x the blank concentration. Reprepare and reanalyze the method blank and all associated samples with results > LOD and < 10x the contaminated blank result. Contact Client if samples cannot be reprepped within hold time.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Calibration blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD. For negative blanks, absolute value < LOD.	Correct problem. Re-prep and reanalyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.	Apply B-flag to all results for specific analyte(s) in all samples associated with the blank.	
LCS containing all analytes to be reported	One per preparatory batch.	Water: Recovery must be within ± 20% of the true value Soil: Recovery must be within vendor supplied limits (varies by lot).	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7).	Recovery must be within ± 20% of the true value	Examine the project-specific DQOs. If the matrix spike falls outside of DoD criteria, additional quality control tests are required to evaluate matrix effects.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix	MSD: Recovery must be within ± 20% of the true value. MSD or sample duplicate: RPD ≤ 20% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply J-flag if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 2

DoD QSM QC REQUIREMENTS

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method of standard additions (MSA)	When matrix interference is confirmed.	NA.	NA.	NA.	Document use of MSA in the case narrative.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

TABLE 3

SUMMARY OF METHOD MODIFICATIONS

Topic	Katahdin SOP CA-611-09	USEPA Method 7471, current revision
Reagents	Stannous chloride dissolved in hydrochloric acid to prevent clogging of mercury analyzer, per instrument manufacturer's recommendation.	Stannous chloride dissolved/suspended in sulfuric acid.
Procedures	Sampling and gas stream switching performed automatically by mercury analyzer.	Sampling and gas stream switching performed manually by analyst.
QC – Calibration Verification	1)Known reference sample (ICV) analyzed daily. 2)Calibration verified after every 10 samples with CCV.	1)Known reference sample analyzed quarterly. 2)Calibration verified after every 20 samples.
QC - Calibration Blanks and Method Blanks	Acceptance Criterion: < PQL	Acceptance criteria: Low enough not to interfere with data quality objectives, or <10% of PQL, or <10% of regulatory limit, or <10% of lowest associated sample

TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 1

EXAMPLE PAGE FROM MERCURY PREPARATION LOGBOOK

Katahdin Analytical Services, Inc.

Metals Preparation Benchsheet

Reagent Information:
HNO3: J41057 HCL: J350-12 H2SO4: N/A
KMNO4: MR 981 K2S2O8: N/A NH2OH-HCl: MR 980

Method: 7471

REVIEWED
DWM 12-7-10
KATAHDIN ANALYTICAL METALS SECTION

Standards/Spiking Information:
1ppm A : MW12757
1ppm B : MW12758
LCSS: 5mL of 1ppm A
Spike(S/P) = 100uL of 1ppm A to 100mL

ICV = 600uL of 1ppm B to 100 mL
S0.2 = 20uL of 1ppm A to 100 mL
S0.5 = 50uL of 1ppm A to 100 mL

S1.0 = 100uL of 1ppm A to 100 mL
S5.0 = 500uL of 1ppm A to 100 mL
S10.0 = 1000uL of 1ppm A to 100 mL

Water Bath ID : A
Thermometer ID : DIG38

Balance ID: BAL08
Digestion Start Time (@ 95 °C): 951420
Digestion End Time(@ 100 °C): 1450

Sample ID	Batch ID	Initial Wt/Vol	Initial Units	Final Vol	Final Units	MX	Meth	Anal.	Date	Initial Color	Initial Texture	Final Color	Final Clarity	Artifacts	Bottle
LCSSAL06HGS0	AL06HGS0	0.1	g	0.10	L	SL	HG	HHH	12/06/2010	N/A	N/A	N/A	N/A		
PBSAL06HGS0	AL06HGS0	0.6	g		L	SL	HG	HHH	12/06/2010	N/A	N/A	N/A	N/A		
SD7209-001	AL06HGS0	0.63	g		L	SL	HG	HHH	12/06/2010						G
SD7209-002	AL06HGS0	0.73	g		L	SL	HG	HHH	12/06/2010						A
SD7209-003	AL06HGS0	0.61	g		L	SL	HG	HHH	12/06/2010						G
SD7209-004	AL06HGS0	0.61	g		L	SL	HG	HHH	12/06/2010						G
SD7209-005	AL06HGS0	0.65	g		L	SL	HG	HHH	12/06/2010						G
SD7209-006	AL06HGS0	0.78	g		L	SL	HG	HHH	12/06/2010						G
SD7209-007	AL06HGS0	0.62	g		L	SL	HG	HHH	12/06/2010						G
SD7209-008	AL06HGS0	0.68	g		L	SL	HG	HHH	12/06/2010						G
SD7209-009	AL06HGS0	0.65	g		L	SL	HG	HHH	12/06/2010						A
SD7421-001	AL06HGS0	0.71	g		L	SL	HG	HHH	12/06/2010						A
SD7421-001P	AL06HGS0	0.71	g		L	SL	HG	HHH	12/06/2010						A
SD7421-001S	AL06HGS0	0.72	g		L	SL	HG	HHH	12/06/2010						A

HHH 12-6-10

QA-067-Revision 1 - 09/23/2010 Digestion performed by: HHH On: 12-8-10 Page: AL024 Revision: 00

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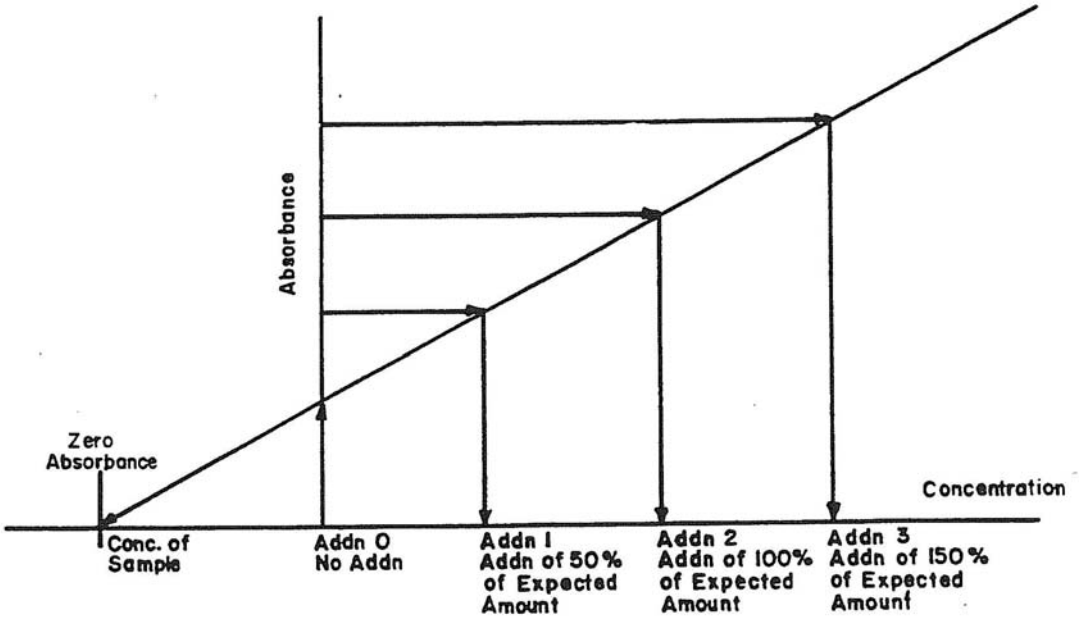
FIGURE 2

STANDARD ADDITIONS PLOT



TITLE: DIGESTION AND ANALYSIS OF SOLID SAMPLES FOR MERCURY BY USEPA METHOD 7471

FIGURE 3

EXAMPLE CERTIFICATE OF ANALYSIS FOR A SOLID REFERENCE MATERIAL

ENVIRONMENTAL RESOURCE ASSOCIATES®
The Industry Standard™

MS1475

DataPacK™
Lot No. D051-540

Trace Metals in Soil
Catalog No. 540

Certification

Method 3050 HNO3, H2O2, HCl Parameter	Total Concentration [1] (mg/Kg)	Certified Value [2] (mg/Kg)	Performance Acceptance Limits™ [3] (mg/Kg)
aluminum	55600*	7870	4630 - 11100
antimony	160	70.5	D.L. - 149
arsenic	316	289	234 - 344
barium	869	211	174 - 247
beryllium	60.9	54.4	45.2 - 63.6
boron	129	91.3	58.8 - 124
cadmium	114	101	82.9 - 119
calcium	9750*	3680	2970 - 4390
chromium	249	224	180 - 268
cobalt	113	101	82.7 - 119
copper	94.9	88.0	73.3 - 103
iron	24400*	15700	6610 - 24900
lead	184	158	129 - 187
magnesium	3780*	2260	1760 - 2750
manganese	703	420	343 - 497
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	69.6	55.5 - 83.7
nickel	137	120	99.1 - 141
potassium	33000*	3000	2200 - 3800
selenium	146	130	101 - 159
silver	127	104	68.9 - 139
sodium	15600*	1080	692 - 1470
strontium	326	113	90.5 - 135
thallium	106	94.0	72.8 - 115
tin	175	149	104 - 194
titanium	3100*	284	116 - 453
vanadium	151	111	85.1 - 137
zinc	311	272	215 - 329

Method 3050 HNO3, H2O2 Parameter	Total Concentration [1] mg/Kg	Certified Value [2] mg/Kg	Performance Acceptance Limits™ [3] mg/Kg
aluminum	55600*	7380	4440 - 10300
antimony	160	75.2	D.L. - 198
arsenic	316	284	225 - 343
barium	869	217	177 - 257
beryllium	60.9	53.6	42.7 - 64.5
boron	129	89.5	58.9 - 120
cadmium	114	103	83.6 - 122
calcium	9750*	3540	2800 - 4270
chromium	249	224	172 - 275
cobalt	113	101	82.0 - 120
copper	94.9	85.5	70.4 - 100
iron	24400*	12500	5480 - 19500
lead	184	162	132 - 192
magnesium	3780*	2160	1650 - 2670
manganese	703	415	330 - 500
mercury	5.32	5.18	3.42 - 6.87
molybdenum	80.2	68.8	52.7 - 84.9
nickel	137	119	98.5 - 140
potassium	33000*	2840	2160 - 3520
selenium	146	135	104 - 166
silver	127	107	49.8 - 164
sodium	15600*	1010	709 - 1310
strontium	326	111	89.0 - 133
thallium	106	99.3	76.8 - 122
tin	175	148	70.6 - 225
titanium	3100*	283	104 - 463
vanadium	151	104	70.5 - 138
zinc	311	275	222 - 328

ADDENDUM

SOP NO CHANGE FORM

KATAHDIN ANALYTICAL SERVICES, INC.
SOP "REVIEW WITH NO CHANGES" FORM

Name of Person Reviewing SOP: Nicholas Tillotson

Review Date: 1/28/13

SOP Number: CA510-07

SOP Title:
TCLP for inorganic and non-volatile organic analytes

THE ABOVE REFERENCED SOP HAS BEEN REVIEWED BY A QUALIFIED AND TRAINED ANALYST OR SUPERVISOR. NO CHANGES ARE REQUIRED TO THE SOP AT THIS TIME.

Department Supervisor Signature: _____ **Date:** 02/26/13

QAO Signature: Leslie Dimond **Date:** 030413

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2005

TESTAMERICA LABORATORIES WEST SACRAMENTO
880 Riverside Parkway
West Sacramento, CA 95605
Douglas Weir Phone: 916 3744 389

ENVIRONMENTAL

Valid To: January 31, 2014 Certificate Number: 2928.01

In recognition of the successful completion of the A2LA evaluation process, (including an assessment of the laboratory's compliance with ISO IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 4.2 of the DoD Quality Systems Manual for Environmental Laboratories) accreditation is granted to this laboratory to perform recognized EPA methods using the following testing technologies and in the analyte categories identified below:

Testing Technologies

Inductively Coupled Plasma (ICP), ICP-Mass Spectroscopy, Atomic Absorption Spectroscopy (flame), Gas Chromatography(GC), GC- Mass Spectroscopy, High Resolution Gas Chromatography/High Resolution Mass Spectroscopy, Liquid Chromatography(LC),LC- Mass Spectroscopy, Ion Chromatography, Spectrophotometry, Misc.- Electronic Probes

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
Metals				
Aluminum	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Antimony	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Arsenic	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Barium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Beryllium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Cadmium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Calcium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Chromium (Total)	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Chromium (Hexavalent)	EPA 7196A	EPA 7196A	-------	EPA 3005A/3010A/3050A
Cobalt	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Copper	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Iron	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Lead	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Magnesium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A

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Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
Manganese	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Mercury	EPA 7470A	EPA 7471A	-------	EPA 3005A/3010A/3050A
Molybdenum	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Nickel	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Potassium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Selenium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Silver	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Sodium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Thallium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Vanadium	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Zinc	EPA 6010B/6020	EPA 6010B/6020	-------	EPA 3005A/3010A/3050A
Nutrients				
Nitrate	EPA 353.2/9056A/300.0	EPA 353.2/9056A/300.0	-------	----------
Nitrate-nitrite	EPA 353.2/ SM4500-NO3 F	EPA 353.2	-------	----------
Nitrite	EPA 353.2/9056A/300.0	EPA 353.2/9056A/300.0	-------	----------
Orthophosphate	EPA 9056A/300.0	EPA 9056A/300.0	-------	----------
Wet Chemistry				
Oil and Grease	EPA 1664A/9070	EPA 1664A/9071	-------	----------
Nitrocellulose	WS-WC-0050	WS-WC-0050	-------	----------
Perchlorate	EPA 6850	EPA 6850	-------	----------
Chloride	EPA 9056A/300.0	EPA 9056A/300.0	-------	----------
Fluoride	EPA 9056A/300.0	EPA 9056A/300.0	-------	----------
Sulfate	EPA 9056A/300.0	EPA 9056A/300.0	-------	----------
			-------	----------
Hazardous Waste Characteristics				
TCLP Extractables	----------	EPA 1311		
TCLP Inorganics	----------	EPA 1311	-------	----------
			-------	----------
Purgeable Organics				
(volatiles)				
Acetone	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Acrolein	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Acrylonitrile	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Allyl Chloride	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Benzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Bromochloromethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Bromodichloromethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Bromoform	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Bromomethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Carbon disulfide	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Carbon tetrachloride	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Chlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
Chloroethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
2-Chloroethyl vinyl ether	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Chloroform	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Chloromethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Chloroprene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Cyclohexane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1-Chlorocyclohexane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Dibromochloromethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2-Dibromo-3-chloropropane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2-Dibromoethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Dibromomethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2-Dichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,3-Dichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,4-Dichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
T-1,4-Dichloro-2-Butene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Dichlorodifluoromethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,1-Dichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2-Dichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,1-Dichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
trans-1,2-Dichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
cis-1,2-Dichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2-Dichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,3-Dichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
2,2-Dichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,1-Dichloropropene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
cis-1,3-Dichloropropene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
trans-1,3-Dichloropropene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,4-Dioxane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Ethylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Ethylmethacrylate	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Hexachlorobutadiene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Hexane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
2-Hexanone (MBK)	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Iodomethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Isobutanol	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Isopropyl Ether	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Methacrylonitrile	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Methyl tert-butyl ether (MTBE)	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Methylene chloride	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Methyl ethyl ketone	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Methyl Methacrylate	EPA 8260B/8260C	EPA 8260B/8260C	-------	----------
4-Methyl-2-pentanone (MIBK)	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Naphthalene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Propionitrile	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,1,1,2-Tetrachloroethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,1,2,2-Tetrachloroethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Tetrachloroethene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Tetrahydrofuran	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
Toluene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2,3-Trichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2,4-Trichlorobenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,1,1-Trichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,1,2-Trichloroethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,1-2-Trichloro-1,2-2-trifluorethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Trichloroethene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Trichlorofluoromethane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2,3-Trichloropropane	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Vinyl acetate	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Vinyl chloride	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
m & p xylene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
o-xylene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Xylenes, Total	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
tert-amyl methyl ether (TAME)	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
tert-butyl alcohol (TBA)	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Ethyl tert-butyl ether (ETBE)	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Bromobenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
n-Butylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
sec-Butylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
tert-Butylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
2-Chlorotoluene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
4-Chlorotoluene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Isopropylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
n-Propylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Styrene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,2,4-Trimethylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
1,3,5-Trimethylbenzene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Oxygenates	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Gasoline Range Organics (GRO)	EPA 8260B/AK101	EPA 8260B/AK101	-------	EPA 5030A/5030B/5035/5035A
p-Isopropyltoluene	EPA 8260B/8260C	EPA 8260B/8260C	-------	EPA 5030A/5030B/5035/5035A
Extractable Organics				
(semivolatiles)				
Acenaphthene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Acenaphthylene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Acetophenone	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-Acetylaminofluorene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4-Aminobiphenyl	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Aniline	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
Anthracene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
7,12-Dimethylbenz(a)anthracene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Aramite	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Benzaldehyde	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Dibenze(a,j)acridine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Benzidine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Benzo(a)anthracene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Benzo(b)fluoranthene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Benzo(k)fluoranthene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Benzo(j)fluoranthene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Benzo(g,h,i)perylene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Benzo(a)pyrene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Benzo(e)pyrene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Benzoic acid	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Benzyl alcohol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Benzyl butyl phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-sec-Butyl-4,6-dinitrophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Bis(2-chloroethoxy) Methane	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Bis(2-chloroethyl) ether	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Bis(2-chloroisopropyl) ether	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Di(2-ethylhexyl) phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4-Bromophenyl phenyl ether	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Carbazole	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
4-Chloroaniline	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4-Chloro-3-methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Hexacloropropene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-Chloronaphthalene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-Chlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4-Chlorophenyl phenyl ether	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Chrysene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Diallate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3510C/3550B/ 3580A
Dibenz(a,h)anthracene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Dibenzofuran	EPA 8270C/8270D	EPA 8270C/8270D	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
1,2-Dichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,3-Dichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,4-Dichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
3,3'-Dichlorobenzidine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,4-Dichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,6-Dichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Diethyl Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,4-Dimethylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Dimethyl Phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Di-n-butyl phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Di-n-octyl phthalate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4,6-Dinitro-2-methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,4-Dinitrophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,4-Dinitrotoluene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,6-Dinitrotoluene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,4-Dioxane	WS-MS-0011	WS-MS-0011	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
1,2-Diphenylhydrazine (as Azobenzene)	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Famphur	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Fluoranthene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Fluorene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Hexachlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Hexachlorobutadiene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Hexachlorocyclopentadiene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Hexachloroethane	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Indeno(1,2,3-c,d)pyrene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Isodrin	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Isophorone	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Isosafrole	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Isosafrole #1	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Isosafrole #2	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Kepone	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Dimethoate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Methapyrilene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Methyl methanesulfonate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Methyl Parathion	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
3-Methylcholanthrene	EPA 8270C/8270D	EPA 8270C/8270D		EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-Methylnaphthalene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
2-Methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
3-Methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4-Methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
3&4-Methylphenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
Naphthalene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
1,4-Naphthoquinone	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1-Chloronaphthalene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1-Methylnaphthalene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1-Naphthylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-Naphthylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
3-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4-Nitroaniline	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4-Nitro-o-toluidine-1-oxide	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
5-Nitro-o-toluidine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Nitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,2-Dinitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,3-Dinitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,4-Dinitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,3,5-Trinitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-Nitrophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
4-Nitrophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitrosodimethylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitrosodi-n-propylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitrosodiphenylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitrosodiethylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitroso-di-n-butylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitrosomethylethylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitrosomorpholine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitrosopyrrolidine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
n-Nitrosopiperidine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
Parathion	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
p-Chorobenzilate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
p-Dimthylaminoazobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Pentachlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Pentaclorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Pentacloronitrobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Pentacloroethane	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Phenacetin	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Phenanthrene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
a,a-Dimethylphenethlamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Phenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Biphenyl	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Diphenylamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
p-Phenylenediamine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2-Picoline	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Phorate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Promamide	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Pyrene	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	EPA 8270C/8270D/WS-MS-006/WS-MS-0008	WS-MS-0006	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A Air: 3542/TO-13A
Pyridine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Safrole	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Sulfotepp	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Disulfotone	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Ethylmethanesulfonate	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Thionazin	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,2,4,5-Tetrachlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
1,2,4-Trichlorobenzene	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
2,4,5-Trichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,3,4,6-Tetrachlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,3,5,6-Tetrachlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
2,4,6-Trichlorophenol	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Diesel Range Organics (DRO)	EPA 8015B/8015C/AK102	EPA 8015B/8015C/AK102	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Residual Range Organics	AK103	AK103	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
o,o,o-TEPT	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
o-Toluidine	EPA 8270C/8270D	EPA 8270C/8270D	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3580A
Dioxins				
2,3,7,8-TeCDD	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B		----------
1,2,3,7,8-PeCDD	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
1,2,3,4,7,8-HxCDD	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
1,2,3,6,7,8-HxCDD	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
1,2,3,7,8,9-HxCDD	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
1,2,3,4,6,7,8-HpCDD	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
OCDD	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
2,3,7,8-TeCDF	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
1,2,3,7,8-PeCDF	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
2,3,4,7,8-PeCDF	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
1,2,3,4,7,8-HxCDF	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
1,2,3,6,7,8-HxCDF	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
1,2,3,7,8,9-HxCDF	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
2,3,4,6,7,8-HxCDF	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
1,2,3,4,6,7,8-HpCDF	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
1,2,3,4,7,8,9-HpCDF	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
OCDF	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
Total TCDD	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
Total PeCDD	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
Total HxCDD	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
Total HeptaCDD	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
Total TCDF	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
Total PeCDF	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
Total HxCDF	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
Total HpCDF	EPA 8280A/8280B/8290 /8290A/1613B	EPA 8280A/8280B/8290 /8290A/1613B	-------	----------
Chemical Warfare Degradates				
1,4-Dithiane	WS-MS-0003	WS-MS-0003	-------	----------
Benzothiazole	WS-MS-0003	WS-MS-0003	-------	----------
p-Chlorophenyl methylsulfide	WS-MS-0003	WS-MS-0003	-------	----------
p-Chlorophenyl methylsulfoxide	WS-MS-0003	WS-MS-0003	-------	----------
p-Chlorophenyl methylsulfone	WS-MS-0003	WS-MS-0003	-------	----------
Chloropicrin	WS-MS-0003	WS-MS-0003	-------	----------
Acetophenone	WS-MS-0003	WS-MS-0003	-------	----------
2-Chloroacetophenone	WS-MS-0003	WS-MS-0003	-------	----------
1,4-Oxathiane	WS-MS-0003	WS-MS-0003	-------	----------
Dimethyl Disulfide	WS-MS-0003	WS-MS-0003	-------	----------
Diisopropylmethylphosphate (DIMP)	WS-LC-0004	WS-LC-0004	-------	----------
Dimethylmethylphosphonate (DMMP)	WS-LC-0004	WS-LC-0004	-------	----------
Ethyl methylphosphonic acid (EMPA)	WS-LC-0004	WS-LC-0004	-------	----------
Isopropyl methylphosphonic acid (IMPA)	WS-LC-0004	WS-LC-0004	-------	----------
Methylphosphonic acid (MPA)	WS-LC-0004	WS-LC-0004	-------	----------
Thiodiglycol (2,2'-Thiodiethanol) (TDG)	WS-LC-0004	WS-LC-0004	-------	----------
Nitroaromatics				
2-Amino-4,6-dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	-------	----------
4-Amino-2,6-dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	-------	----------
3,5-Dinitroaniline	EPA 8330B	EPA 8330B	-------	----------
1,3-Dinitrobenzene	EPA 8330A/8330B	EPA 8330A/8330B	-------	----------
2,4-Dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	-------	----------
2,6-Dinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	-------	----------
Ethylene glycol dinitrate	EPA 8330B	EPA 8330B	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
Glycerol trinitrate (Nitroglycerin)	EPA 8330B	EPA 8330B	-------	----------
Hexahydro-1,3,5-trinitro-1,3,5-triazine (Hexogen)	EPA 8330A/8330B	EPA 8330A/8330B	-------	----------
Methyl-2,4,6-trinitrophenylnitramine	EPA 8330A/8330B	EPA 8330A/8330B	-------	----------
Nitrobenzene	EPA 8330A/8330B	EPA 8330A/8330B	-------	----------
2-Nitrotoluene (o-Nitrotoluene)	EPA 8330A/8330B	EPA 8330A/8330B	-------	----------
3-Nitrotoluene (m-Nitrotoluene)	EPA 8330A/8330B	EPA 8330A/8330B	-------	----------
4-Nitrotoluene (p-Nitrotoluene)	EPA 8330A/8330B	EPA 8330A/8330B	-------	----------
Octahydro-1,3,5,7-tetranitro1,3,5,7-tetracine (Octogen)	EPA 8330A/8330B	EPA 8330A/8330B	-------	----------
Picric acid	EPA 8330B	EPA 8330B	-------	----------
Pentaerythritol tetranitrate	EPA 8330B	EPA 8330B	-------	----------
1,3,5-Trinitrobenzene	EPA 8330A/8330B	EPA 8330A/8330B	-------	----------
2,4,6-Trinitrotoluene	EPA 8330A/8330B	EPA 8330A/8330B	-------	----------
Nitroguanidine	WS-LC-0010	WS-LC-0010	-------	----------
Nitrosamines				
N-Nitrosodimethylamine (NDMA)	WS-MS-0012	WS-MS-0012	-------	----------
Perfluoro Compounds				
Perfluorooctanoic acid	WS-LC-0020	WS-LC-0020	-------	----------
Perfluorooctane sulfonate	WS-LC-0020	WS-LC-0020	-------	----------
Pesticides/PCBs				
Aldrin	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
a-BHC	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
b-BHC	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
d-BHC	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
g-BHC (Lindane)	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
a-Chlordane	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
g-Chlordane	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Oxy-Chlordane	EPA 1699	EPA 1699		EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
4,4'-DDD	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
2,4'-DDD	EPA 1699	EPA 1699		EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
4,4'-DDE	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
2,4'-DDE	EPA 1699	EPA 1699		EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
4,4'-DDT	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
2,4'-DDT	EPA 1699	EPA 1699		EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Dieldrin	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Endosulfan I	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Endosulfan II	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Endosulfan sulfate	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Endrin	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Endrin aldehyde	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Endrin ketone	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Heptachlor	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Heptachlor epoxide	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Hexachlorobenzene	EPA 1699	EPA 1699		EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Methoxychlor	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Cis-Nonachlor	EPA 1699	EPA 1699		EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Trans-Nonachlor	EPA 1699	EPA 1699		EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Toxaphene	EPA 8081A/8081B/EPA 1699	EPA 8081A/8081B/EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Mirex	EPA 1699	EPA 1699	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
Chlordane (technical)	EPA 8081A/8081B	EPA 8081A/8081B	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A
PCB (Aroclors)				
PCB-1016	EPA 8082/8082A	EPA 8082/8082A	-------	EPA 3500B/3500C/3510C/ 3550B/3550C/3620B/3660A/ 3620B/3665A
PCB-1221	EPA 8082/8082A	EPA 8082/8082A	-------	EPA 3500B/3500C/3510C/
PCB-1232	EPA 8082/8082A	EPA 8082/8082A	-------	3550B/3550C/3620B/3660A/
PCB-1242	EPA 8082/8082A	EPA 8082/8082A	-------	3620B/3665A
PCB-1248	EPA 8082/8082A	EPA 8082/8082A	-------	EPA 3500B/3500C/3510C/
PCB-1254	EPA 8082/8082A	EPA 8082/8082A	-------	3550B/3550C/3620B/3660A/
PCB-1260	EPA 8082/8082A	EPA 8082/8082A	-------	3620B/3665A

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
PCB-1262	EPA 8082/8082A	EPA 8082/8082A	-------	EPA 3500B/3500C/3510C/
PCB-1268	EPA 8082/8082A	EPA 8082/8082A	-------	3550B/3550C/3620B/3660A/
PCB (congeners)				
PCB 1 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 2 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 3 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 4 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 5 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 6 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 7 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 8 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 9 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 10 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 11 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 12 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 13 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 14 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 15 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 16 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 17 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 18 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 19 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 20 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 21 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 22 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 23 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 24 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 25 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
PCB 26 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 27 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 28 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 29 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 30 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 32 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 31 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 33 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 34 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 35 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 36 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 37 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 38 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 39 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 40 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 41 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 42 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 43 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 44 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 45 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 46 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 47 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 48 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 49 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 50 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 51 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 52 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
PCB 53 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 54 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 55 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 56 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 57 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 58 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 59 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 60 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 61 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 62 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 63 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 64 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 65 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 66 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 67 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 68 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 69 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 70 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 71 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 72 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 73 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 74 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 75 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 76 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 77 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 78 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 79 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
PCB 80 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 81 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 82 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 83 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 84 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 85 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 86 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 87 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 88 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 89 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 90 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 91 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 92 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 93 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 94 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 95 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 96 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 97 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 98 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 99 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 100 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 101 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 102 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 103 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 104 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 105 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 106 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
PCB 108 (BZ)/107 (IUPAC)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 109 (BZ)/108 (IUPAC)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 107 (BZ)/109 (IUPAC)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 110 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 111 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 112 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 113 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 114 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 115 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 116 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 117 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 118 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 119 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 120 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 121 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 122 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 123 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 124 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 125 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 126 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 127 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 128 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 129 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 130 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 131 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 132 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 133 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
PCB 134 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 135 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 136 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 137 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 138 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 139 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 140 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 141 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 142 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 143 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 144 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 145 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 146 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 147 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 148 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 149 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 150 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 151 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 152 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 153 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 154 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 155 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 156 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 157 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 158 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 159 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 160 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
PCB 161 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 162 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 163 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 164 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 165 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 166 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 167 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 168 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 169 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 170 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 171 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 172 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 173 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 174 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 175 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 176 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 177 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 178 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 179 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 180 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 181 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 182 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 183 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 184 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 185 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 186 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 187 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
PCB 188 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 189 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 190 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 191 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 192 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 193 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 194 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 195 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 196 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 197 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 198 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 201 (BZ)/199 (IUPAC)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 199 (BZ)/200 (IUPAC)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 200 (BZ)/201 (IUPAC)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 202 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 203 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 204 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 205 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 206 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 207 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 208 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
PCB 209 (BZ)	EPA 1668A mod/1668C mod	EPA 1668A mod/ 1668C mod	-------	----------
Hormones,Steroids, Pharmaceuticals and Disinfection Byproducts				
Acetominophen	WS-LC-0024	----------	-------	----------
Atenolol	WS-LC-0024	----------	-------	----------
Azithromycin	WS-LC-0024	----------		
Carbadox	WS-LC-0024	----------	-------	----------
Carbamazepine	WS-LC-0024	----------	-------	----------

Parameter/Analyte	Non potable Water	Solid Hazardous Waste	Air	Associated Prep Methods
Clarithromycin	WS-LC-0024	----------		
Diazepam	WS-LC-0024	----------	-------	----------
Digoxigenin	WS-LC-0024	----------	-------	----------
Digoxin	WS-LC-0024	----------		
Diphenylhydramine	WS-LC-0024	----------	-------	----------
Fluoxetine	WS-LC-0024	----------		
Flumequine	WS-LC-0024	----------	-------	----------
Gemfibrozil	WS-LC-0024	----------		
Ibuprofen	WS-LC-0024	----------	-------	----------
Naproxen	WS-LC-0024	----------	-------	----------
Ormetoprim	WS-LC-0024	----------	-------	----------
Penicillin G	WS-LC-0024	----------		
Sulfachloropyridazine	WS-LC-0024	----------	-------	----------
Sulfadiazine	WS-LC-0024	----------	-------	----------
Sulfamethizole	WS-LC-0024	----------	-------	----------
Sulfamethoxazole	WS-LC-0024	----------	-------	----------
Sulfathiazole	WS-LC-0024	----------	-------	----------
Thiabendazole	WS-LC-0024	----------		
Trimethoprim	WS-LC-0024	----------		
Tris (2-chloro-2-propyl)phosphate (TCPP)	WS-LC-0024	----------	-------	----------
Warfarin	WS-LC-0024	----------	-------	----------

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In recognition of the successful completion of the A2LA evaluation process that includes an assessment of the laboratory's compliance with ISO/IEC 17025:2005, the 2003 NELAC Chapter 5 Standard, and the requirements of the Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in version 4.2 of the DoD Quality System Manual for Environmental Laboratories (QSM); accreditation is granted to this laboratory to perform recognized EPA methods as defined on the associated A2LA Environmental Scope of Accreditation. This accreditation demonstrates technical competence for this defined scope and the operation of a laboratory quality management system (*refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009*).

AMERICAN ASSOCIATION FOR LABORATORY ACCREDITATION
CORPORATE SEAL 1978
DISTRICT OF COLUMBIA
A2LA

Presented this 27th day of March 2012.

President & CEO
For the Accreditation Council
Certificate Number 2928.01
Valid to January 31, 2014
Revised June 1, 2012

For the tests or types of tests to which this accreditation applies, please refer to the laboratory's Environmental Scope of Accreditation.

Title: Analysis of Samples for Polychlorinated Dioxins and Furans by HRGC/HRMS

[Methods 8290, 8290A & TO-9A]

Approvals (Signature/Date):

Robert Hrabak — 4/2/13
Technical Manager

Joe Schairer — 4/3/13
Health & Safety Manager / Coordinator

Gary Costley — 4-2-13
Quality Assurance Manager

Karla Buechler — 4/4/13
Laboratory Director

1. SCOPE AND APPLICATION

1.1.1. This method provides procedures for the detection and quantitative measurement of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD), polychlorinated dibenzo-p-dioxins (tetra- through octachlorinated homologs; PCDDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologs; PCDFs) in a variety of environmental matrices at part-per-trillion (ppt) concentrations by SW 846 Method 8290 and 8290A. The analytical method calls for the use of high-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. An optional method for reporting the analytical results using a 2,3,7,8-TCDD toxicity equivalency factor (TEF) is also described. Table 1 lists the various sample types covered by this analytical protocol, the 2,3,7,8-TCDD-based method calibration limits and other pertinent information.

1.2. The sensitivity of this method is dependent upon the level of interferences within a given matrix.

1.3. This method is designed for use by analysts who are experienced with residue analysis and skilled in high-resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS).

1.4. Samples containing concentrations of specific congeners (PCDDs and PCDFs) that are greater than the calibration limit should be analyzed by a protocol designed for such concentrations, such as 8280A/B.

1.5. When undertaking projects for Department of Defense (DoD) the relevant criteria in QA Policy WS-PQA-021 “DoD QSM and AFCEE QAPP Implementation” must be checked and incorporated.

2. SUMMARY OF METHOD

2.1. This procedure uses matrix-specific extraction, analyte-specific cleanup, and high-resolution capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS) techniques. Sample preparation is addressed in WS-IDP-0005.

2.2. One to two µL of the concentrated extract are injected into an HRGC/HRMS system capable of performing selected ion monitoring at resolving powers of at least 10,000 (10 percent valley definition).

2.3. The identification of ten of the 2,3,7,8-substituted congeners (Table 3), for which a ^{13}C-labeled standard is included as a spiked compound, is based on their elution at their exact retention time (-1 to +3 seconds from the respective isotope dilution analyte or internal standard signal) and simultaneous detection of the two most abundant ions in

the molecular ion region. All other identified PCDD/PCDF congeners are identified by their RRT's based on the daily CCV standard, and the simultaneous detection of the two most abundant ions in the molecular ion region. Confirmation is based on a comparison of the ratio of the integrated ion abundance of the molecular ion species to their theoretical abundance ratio.

2.4. Quantification of the individual congeners, total PCDDs and total PCDFs is achieved in conjunction with the establishment of a multipoint (five points) calibration curve for each homolog, during which each calibration solution is analyzed once.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

3.3. Polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs): compounds (Figure 1) that contain from one to eight chlorine atoms. The seventeen 2,3,7,8-substituted PCDDs and PCDFs are shown in Table 3. The number of isomers at different chlorination levels is shown in Table 4.

3.4. Homologous series: Defined as a group of chlorinated dibenzodioxins or dibenzofurans having a specific number of chlorine atoms.

3.5. Isomer: Chemical compounds that contain the same number of atoms of the same elements, but differ in structural arrangement and properties. For example, 1,2,3,4-TCDD and 2,3,7,8-TCDD are different structural isomers.

3.6. Congener: Any isomer of any homologous series.

3.7. Isotope Dilution Analyte: An isotope dilution analyte is a ^{13}C-labeled analog of a congener chosen from the compounds listed in Table 3. Isotope dilution analytes are added to all samples including method blanks and quality control samples before extraction, and they are used to quantitate the concentration of the analytes. Nine isotope dilution analytes are used in this method. There is one for each of the dioxin and furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight. Additional isotope dilution analytes may be added to act as retention time references, but they are not used for quantitation.

3.8. Internal Standard: Two internal standards are used to determine the percent recoveries for the isotope dilution analytes. The ^{13}C-1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated isotope dilution analytes while ^{13}C-1,2,3,7,8,9-HxCDD is used to determine the recovery of the hexa-, hepta- and

octachlorinated isotope dilution analytes. ^{13}C-1,2,3,7,8,9-HxCDD also acts as a retention time reference for the unlabeled analog present in sample extracts. They are added to the final sample extract before HRGC/HRMS instrument analysis.

3.9. Estimated Detection Limit (EDL)/ Estimated Quantiation Limit (EQL): The sample specific estimated detection limit (EDL/EQL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background noise level.

3.10. Estimated Maximum Possible Concentration (EMPC): The calculated concentration of a signal having the same retention time as a PCDD/PCDF congener, but which does not meet the other qualitative identification criteria defined in the method.

4. INTERFERENCES

4.1. Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferents under the conditions of analysis by running laboratory method blanks. Analysts shall not use PVC gloves.

4.2. The use of high-purity reagents and solvents helps minimize interference problems. Purification of solvents by distillation in all-glass systems may be necessary.

4.3. Re-use of glassware is to be minimized to avoid the risk of contamination.

4.4. Interferents co-extracted from the sample will vary considerably from matrix to matrix. PCDDs and PCDFs are often associated with other interfering chlorinated substances such as polychlorinated biphenyls (PCBs), polychlorinated diphenyl ethers (PCDPEs), polychlorinated naphthalenes, and polychlorinated xanthenes that may be found at concentrations several orders of magnitude higher than the analytes of interest. Retention times of target analytes must be verified using reference standards. These values must correspond to the retention time windows established. While certain clean-up techniques are provided as part of this method, unique samples may require additional cleanup steps to achieve lower detection limits.

4.5. A high-resolution capillary column (60m DB-5) is used to resolve as many PCDD and PCDF isomers as possible. However, no single column is known to resolve all isomers. The DB-225 column is used for the quantitation of 2,3,7,8-TCDF when 2,3,7,8-TCDF on the DB-5 column is detected.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the Sacramento Addendum to the Corporate EH&S

Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toed, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. The effluents of sample splitters for the gas chromatograph and roughing pumps on the HRGC/HRMS system should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohols.

5.1.2. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile or similar gloves must be used. Latex gloves may be used for methanol.

5.1.3. Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.4. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Iso-octane	Flammable Irritant	None established	Inhalation of vapors may cause nausea, headache, dizziness, loss of consciousness, irritation to upper respiratory tract, pain in throat and nose, coughing, wheezing, shortness of breath.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Tetradecane	Irritant	None established	Inhalation of vapors may cause difficulty breathing, headache, intoxication and central nervous system damage.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

6.1. Preventive and routine maintenance is described in the 'Schedule of Routine Maintenance' in the QAM.

6.2. High-Resolution Gas Chromatograph/High-Resolution Mass Spectrometer/Data System (HRGC/HRMS/DS).

6.2.1. Capable of collecting, recording and storing MS data. The VG70 and Autospec Ultima systems utilize Opus version 3.6 software and the Autospec Premiere system utilizes MassLynx version 4.1 software.

6.2.2. The GC must be equipped for temperature programming. All required accessories must be available, such as syringes, gases, and capillary columns. The GC injection port must be designed for capillary columns. The use of splitless injection techniques is recommended. The use of a moving needle

injection port is also acceptable. When using the method described in this protocol, a 2-µL injection volume is used consistently (i.e., the injection volumes for all extracts, blanks, calibration solutions and the performance check samples are 2 µL). 1 µL injections are allowed; however, laboratories are encouraged to remain consistent throughout the analyses by using the same injection volume at all times on a given HRGC/HRMS/DS.

6.2.3. Gas Chromatograph/Mass Spectrometer (GC/MS) Interface - The GC/MS interface components should withstand 350° C. The interface must be designed so that the separation of 2,3,7,8-TCDD from the other TCDD isomers achieved in the gas chromatographic column is not appreciably degraded. Cold spots or active surfaces (adsorption sites) in the GC/MS interface can cause peak tailing and peak broadening. It is recommended that the GC column be fitted directly into the mass spectrometer ion source without being exposed to the ionizing electron beam. Graphite ferrules should be avoided in the injection port because they may adsorb the PCDDs and PCDFs. Vespel® or equivalent ferrules are recommended.

6.2.4. Mass Spectrometer - The static resolving power of the instrument must be maintained at a minimum of 10,000 (10 percent valley). The mass spectrometer must be operated in a selected ion monitoring (SIM) mode with a total cycle time (including the voltage reset time) of one second or less.

6.2.5. Data System - A dedicated data system is employed to control the rapid multiple ion monitoring process and to acquire the data. Quantification data (peak areas or peak heights) and SIM traces (displays of intensities of each ion signal being monitored including the lock-mass ion as a function of time) must be acquired during the analyses and stored. Quantifications may be reported based upon computer-generated peak areas or upon measured peak heights (chart recording). The data system must be capable of acquiring data for a minimum of 10 ions in a single scan. It is also recommended to have a data system capable of switching to different sets of ions (descriptors) at specified times during an HRGC/HRMS acquisition. The data system should be able to provide hard copies of individual ion chromatograms for selected gas chromatographic time intervals. It should also be able to acquire mass-spectral peak profiles and provide hard copies of peak profiles to demonstrate the required resolving power. The data system should also permit the measurement of noise on the base line.

6.3. GC Column

6.3.1. Due to poor separation of 2,3,7,8-TCDF from other TCDF isomers on the 60 m DB-5 column, a 30M DB-225 is used to quantitate 2,3,7,8-TCDF. This column is used when 2,3,7,8-TCDF is detected.

6.3.2. In order to have an isomer-specific determination for 2,3,7,8-TCDD and to allow the detection of OCDD/OCDF within a reasonable time interval in one HRGC/HRMS analysis, the 60-m DB-5 fused-silica capillary column is recommended. At the beginning of each 12-hour period during which samples are analyzed and after tuning, acceptable compound separation on the GC column must be demonstrated through the analysis of a column performance check solution. Operating conditions known to produce acceptable results with the recommended column are shown in Table 7.

7. REAGENTS AND STANDARDS

7.1. Solvents

7.1.1. High-purity, distilled-in-glass or highest available purity: methylene chloride, hexane, methanol, tetradecane, isooctane, toluene, and acetone.

7.2. All calibration, daily isotope dilution analyte, daily clean up internal standards, and daily spiking solutions are stable for one year from preparation. After 1 year, solutions may be re-verified. The re-verified solution may be used for an additional year, or until there is evidence of compound degradation or concentration. The re-verification must be performed using an unexpired, not previously re-verified solution from a second lot or second vendor.

7.2.1. Sealed ampules may be used until the manufacturer's expiration date is exceeded. If no expiration date is provided, then the expiration date will be 10 years from the date the ampule is opened. The solvent level should be monitored prior to each use to assure there has been no concentration of the standard over time.

7.3. Calibration Solutions

7.3.1. High-Resolution Concentration Calibration Solutions (Table 5) - Five tetradecane solutions containing unlabeled (totaling 17) and carbon-labeled (totaling 16) PCDDs and PCDFs at known concentrations are used to calibrate the instrument. The concentration ranges are homolog dependent, with the lowest values associated with the tetra chlorinated dioxins and furans (0.5 pg/µL) and the highest for the octachlorinated congeners (2000 pg/µL).

7.3.2. Individual isomers that make up the high-resolution concentration calibration solutions are obtained from commercial sources and prepared in the laboratory. These standards are traceable back to EPA-supplied standard solutions.

7.3.3. Store the calibration solutions in appropriate containers and at room temperature in the dark.

7.3.4. Standards for method 8290A require storage at ≤ 6°C.

7.4. GC Column Performance Check Solution

7.4.1. This solution contains the first and last eluting isomers for each homologous series from tetra- through hepta-chlorinated congeners. The solution also contains a series of other TCDD isomers for the purpose of documenting the chromatographic resolution. The ^{13}C-2,3,7,8-TCDD is also present. The laboratory is required to use tetradecane as the solvent and adjust the volume so that the final concentration does not exceed 100 pg/µL per congener. Table 8 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution for the DB-5 column.

7.4.2. For the DB-225 column, the column performance check solution contains a series of TCDF isomers in addition to the 2,3,7,8-TCDF. The solution is injected and evaluated at the start of each analytical sequence on the DB-225 column to ensure that 2,3,7,8-TCDF is resolved from its closest eluting isomers with a baseline-to-valley ratio of ≤ 25%. Table 8 summarizes the qualitative composition (minimum requirement) of this performance evaluation solution on for the DB-225 column.

7.5. Field Surrogate Solution (air matrices)

7.5.1. This solution contains one ^{37}Cl labeled analog (for Method TO-9/TO-9A) or one ^{37}Cl and four ^{13}C labeled analogs (for Methods 23 and/or 0023A) at the nominal concentration indicated in Table 2. It is used to assess sample collection and recovery procedures.

7.6. Sample Fortification Solution (Isotope dilution analyte)

7.6.1. This isooctane (or toluene) solution contains the nine isotope dilution analytes at the nominal concentrations that are listed in Table 2. The solution contains at least one carbon-labeled standard for each homologous series, and it is used to measure the concentrations of the native substances. (Note that ^{13}C-OCDF is not present in the solution.)

7.7. Internal Standard Solution

7.7.1. This tetradecane solution contains two internal standards (^{13}C-1,2,3,4-TCDD and ^{13}C-1,2,3,7,8,9-HxCDD). An appropriate volume of this solution will be spiked into each sample extract before the final concentration step and HRGC/HRMS analysis.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. The sample collection, shipping, handling, and chain-of-custody procedures are not described in this document. Sample collection personnel will, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory. The analyst should make a judgment, based on the appearance of the sample, regarding the necessity for additional mixing. If the sample is clearly non-homogeneous, the entire contents should be transferred to a glass or stainless steel pan for mixing with a stainless steel spoon or spatula before removal of a sample portion for analysis.

8.2. Grab and composite samples must be collected in glass containers.

8.3. Ambient air samples are collected on a Quartz Fiber Filter followed by a glass sleeve containing a polyurethane foam plug.

8.4. Samples from stationary sources are collected on glass or quartz fiber filters and XAD-2 Resin. (See WS-ID-0009 for sample preparation procedures).

8.5. Conventional sampling practices must be followed. Do not rinse the bottle with sample before collection. Sampling equipment must be free of potential sources of contamination.

8.6. With the exception of the fish tissues, which must be stored at - 20°C, all samples should be stored at 4°C ± 2, extracted within 30 days and completely analyzed within 45 days of collection. The 30 day hold time is recommended. PCDDs and PCDFs have demonstrated stability for greater than one year.

8.7. All extracts must be stored capped, in the dark, at room temperature (approximately 21°C to 28°C). All extracts for method 8290A must be stored capped at $\leq 6^{o}$C.

9. QUALITY CONTROL

9.1. One method blank (MB) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory matrix (reagent water, Ottawa sand, sodium sulfate, PUF, XAD, filter, etc.) processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. The method blank contains a PUF plug, XAD, or filter prepared from the same batch as the field samples whenever possible for air samples.

Certain programs, such as DOD, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the lower calibration limit.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.1.1. The method blank must be spiked prior to extraction with the same amount of ^{13}C-labeled isotope dilution analytes as added to samples.

9.1.2. If method blank contamination is present, check solvents, reagents, fortification solutions, apparatus and glassware to locate and eliminate the source of contamination before any further samples are extracted and analyzed.

9.1.2.1. OCDD is a ubiquitous laboratory contaminant. A method blank and the associated samples are deemed acceptable if the OCDD concentration is <5x the specified reporting limit. Flag data appropriately. The analyst is expected to investigate and eliminate potential sources of systematic contamination.

9.1.2.2. If a target analyte is detected in the blank but the associated samples are ND (not detected), then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.2.3. If a target analyte is detected in the blank, but the concentration of the contaminant in the samples >10x the blank concentration, then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.3. If new batches of reagents or solvents contain interfering contaminants, purify or discard them.

9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, Ottawa sand, sodium sulfate, PUF, XAD, etc.) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See policy WS-PQA-003 for specific acceptance criteria. When associated with PUF samples, the LCS should contain a PUF plug prepared from the same batch as the field samples whenever possible.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.2.1. A LCS is deemed acceptable if control analytes are above upper control limits and the associated samples are ND, unless otherwise specified by the client. Note any actions in the narrative.

9.3. The assessment of matrix effects on method performance, as required by NELAP, is met in Method 8290 and 8290A, as in all isotope dilution techniques, with the use of isotopically labeled compounds. These isotopically labeled compounds are analogs of target analytes and are spiked into each sample. Therefore, matrix effects on method performance may be judged by the recovery of these analogs. Sample analysis acceptance is controlled by the performance of these analogs in each sample. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair are extracted at the client's request only. Method 8290A does not address analysis of MS/MSD. An exception to this rule is a batch containing South Carolina samples for Method 8290. These batches must have an MS/MSD prepared. However, South Carolina requires Method 8290A after December 31, 2008. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. When requested by the client, the MS/MSD pair shall be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, the LCS, the selected field sample, and the MS/MSD may be required after evaluation and review. Matrix Spike/ Matrix Spike Duplicates are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. An LCS/LCSD may be extracted to show precision of the extraction and analysis process.

9.3.1. Matrix Spike (MS): A sample, which is spiked with a known amount of the matrix spike fortification solution prior to the extraction step. The recoveries of the matrix spike compounds are determined; they are used to estimate the effect of the sample matrix upon the analytical methodology.

9.3.2. Matrix Spike Duplicate (MSD): A second portion of the same sample as used in the matrix spike analysis and which is treated like the matrix spike sample.

9.3.3. Locate the sample for the MS and MSD analyses (the sample may be labeled "double volume").

9.3.4. Add an appropriate volume of the matrix spike fortification solution, adjusting the fortification level as specified in Table 1, under IS Spiking Levels.

9.3.5. Analyze the MS and MSD samples as described in Section 11.

9.3.6. The results obtained from the MS and MSD samples (percent recovery and concentrations of 2,3,7,8-substituted PCDDs/PCDFs) should agree within 20 percent relative difference. Report all results and flag outliers.

9.3.7. Isotope dilution analyte recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any isotope dilution analyte is less than 10:1.

9.4. Duplicates

9.4.1. Upon client request, duplicates may be processed. Locate the sample specified for duplicate analysis, and prepare and analyze a second 10-g soil or sediment sample portion or 1-L water sample, or an appropriate amount of the type of matrix under consideration. Duplicate samples are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. A duplicate injection of a sample extract may be performed to display instrument precision.

9.4.1.1. The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference. Report all results and flag outliers.

9.4.2. Isotope dilution analyte recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any isotope dilution analyte is less than 10:1.

9.5. Surrogate/Clean Up Internal Standard

A surrogate compound may be spiked into all air media samples prior to collection. For all other matrices, a clean up internal standard is spiked following extraction and just prior to cleanup, in order to monitor relative loss of isotope dilution analyte during both extraction and cleanup.

9.6. Isotope Dilution Analytes

9.6.1. Isotope dilution analytes must be spiked into all samples, QC samples, and included in all calibrations.

9.6.2. For each sample and QC aliquot, calculate the percent recovery. The percent recovery should be between 40 percent and 135 percent for all nine isotope dilution analytes.

9.6.3. A low or high percent recovery for a blank does not require discarding the analytical data but it may indicate a potential problem with future analytical data. Isotope dilution analyte recoveries are flagged if they are outside the

recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any isotope dilution analyte is less than 10:1.

9.7. Recommended Corrective Actions and Troubleshooting Steps

- Verify satisfactory instrument performance.
- If possible, verify that no error was made while weighing the sample portions.
- Review the analytical procedures with the performing laboratory personnel.

10. CALIBRATION

Calibration and Standardization requires a check of mass resolution (tuning), a check of chromatographic resolution, a verification of switching times (i.e. descriptors), and a calibration curve verification.

10.1. For details of the calculations used to generate the regression equations, and how to use the factors generated by these equations, refer to SOP CA-Q-S-005 "Calibration Curves (General)".

10.2. Tuning (Mass Resolution Check)

10.2.1. The mass spectrometer must be operated in the electron ionization mode. A static resolving power of at least 10,000 (10 percent valley definition) must be demonstrated at appropriate masses before any analysis is performed. Corrective actions must be implemented whenever the resolving power does not meet the requirement.

10.2.2. Chromatography time for PCDDs and PCDFs exceeds the long-term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm (e.g., 5 ppm in mass) can have serious adverse effects on instrument performance. Therefore, a mass-drift correction is mandatory. To that effect, it is recommended to select a lock-mass ion from the reference compound (PFK is recommended) used for tuning the mass spectrometer. The selection of the lock-mass ion is dependent on the masses of the ions monitored within each descriptor. Table 6 offers some suggestions for the lock-mass ions. However, an acceptable lock-mass ion at any mass between the lightest and heaviest ion in each descriptor can be used to monitor and correct mass drifts. The level of the reference compound (PFK) metered into the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the most intense selected lock-mass ion signal (regardless of the descriptor number) does not exceed 10 percent of the full-scale deflection for a given set of detector parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

NOTE: Excessive PFK (or any other reference substance) may cause noise problems and contamination of the ion source resulting in downtime for source cleaning.

10.2.3. By using a PFK molecular leak, tune the instrument to meet minimum required resolving power of 10,000 (10 percent valley) at m/z 304.9824 (PFK) or any other reference signal close to m/z 303.9016 (from TCDF). Verify that the exact mass of m/z 380.9760 (PFK) is within 5 ppm of the required value. Note that the selection of the low- and high-mass ions must be such that they provide the largest voltage jump performed in any of the five mass descriptors (Table 6).

10.2.4. Documentation of the instrument resolving power must then be accomplished by recording the peak profile of the high-mass reference signal (m/z 380.9760). The minimum resolving power of 10,000 must be demonstrated on the high-mass ion while it is transmitted at a lower accelerating voltage than the low-mass reference ion, which is transmitted at full sensitivity. The format of the peak profile representation (Figure 3) must allow manual determination of the resolution, i.e., the horizontal axis must be a calibrated mass scale (amu or ppm per division). The result of the peak width measurement (performed at 5 percent of the maximum, which corresponds to the 10-percent valley definition) must appear on the hard copy and cannot exceed 100 ppm at m/z 380.9760 (or 0.038 amu at that particular mass).

10.3. Performance Checks

10.3.1. At the beginning of each 12-hour period during which samples are to be analyzed, aliquots of the 1) GC column performance check solution and 2) high-resolution concentration calibration solution No. 4 (HRCC-4) shall be analyzed to demonstrate adequate GC resolution and sensitivity, response factor reproducibility, and mass range calibration, and to establish the PCDD/PCDF retention time windows. (Note: A HRCC-3 or HRCC-5 may be acquired to meet the requirement of #2 above. This is to provide documentation of consistency for varying concentration levels, and to meet NELAC requirements). A mass resolution check shall also be performed to demonstrate adequate mass resolution using an appropriate reference compound (PFK is recommended). If the required criteria are not met, remedial action must be taken before any samples are analyzed. The mass resolution check will be taken at the beginning and completion of an analytical sequence. An analytical sequence may contain one or more 12 hour periods.

10.3.1.1. Method blanks or solvent blanks are used to demonstrate that the analytical system is free of contamination after the analysis of calibration standards or high level samples. The blank must demonstrate that the system has returned to appropriate background levels prior to continued analysis.

10.3.2. At a minimum, the ions listed in Table 6 for each of the five SIM descriptors

must be monitored. Note that the PeCDF masses (M+2 & M+4) are also monitored in the first descriptor. This is because the first PeCDF isomer elutes closely to the final tetra isomer. The selection (Table 6) of the molecular ions M and M+2 for ^{13}C-HxCDF and ^{13}C-HpCDF rather than M+2 and M+4 (for consistency) is to eliminate, even under high-resolution mass spectrometric conditions, interferences occurring in these two ion channels for samples containing high levels of native HxCDDs and HpCDDs. It is important to maintain the same set of ions for both calibration and sample extract analyses. The recommended mass spectrometer tuning conditions are based on the groups of monitored ions shown in Table 6.

10.3.2.1. The GC column performance check mixture, high-resolution concentration calibration solutions, and the sample fortification solutions may be obtained from the EMSL-CIN. However, if not available from the EMSL-CIN, standards can be obtained from other sources, and solutions can be prepared in the laboratory. Concentrations of all solutions containing 2,3,7,8-substituted native PCDDs/PCDFs, must be verified by comparison with second-source standard solutions.

10.4. Initial Calibration

Initial calibration is required before any samples are analyzed for PCDDs and PCDFs. Initial calibration is also required if any routine calibration (Section 10.5) does not meet the required criteria listed in Section 10.6.

10.4.1. Five high-resolution concentration calibration solutions, listed in Table 5, must be used for the initial calibration.

10.4.2. Tune the instrument with PFK.

10.4.3. Inject 1 or 2 µL of the GC column performance check solution and acquire SIM mass spectral data as described earlier in Section 6.1.3. The total cycle time must be ≤ 1 second. This is analyzed prior to a calibration curve to set descriptor windows only and may not otherwise be documented. The laboratory must not analyze samples until it is demonstrated and documented that the criterion listed in Section 13.1 is met.

10.4.3.1. Select the injection volume based upon the expected target analyte concentration, or expected matrix interferences.

10.4.3.2. The same injection volume must be used for all samples, QC, and standards.

10.4.4. By using the same GC and mass spectrometer conditions that produced acceptable results with the column performance check solution, analyze a 1 or

2-µL portion of each of the five concentration calibration solutions once with the following mass spectrometer operating parameter.

10.4.4.1. The total cycle time for data acquisition must be < 1 second. The total cycle time includes the sum of all dwell times and voltage reset times.

10.4.4.2. Acquire SIM data for all the ions listed in the five descriptors of Table 6.

10.4.4.3. The ratio of integrated ion current for the ions appearing in Table 9 (homologous series quantification ions) must be within the indicated control limits (set for each homologous series).

10.4.4.4. The ratio of integrated ion current for the ions belonging to the ^{13}C labeled isotope dilution analytes and internal standards must be within the control limits stipulated in Table 9.

NOTE: Section 10.4.3 requires that ion ratios be within the specified control limits simultaneously in one run. It is the laboratory's responsibility to take corrective action if the ion abundance ratios are outside the limits.

10.4.5. For each SICP and for each GC signal corresponding to the elution of a target analyte and of its labeled standards, the signal-to-noise ratio (S/N) must be better than or equal to 10. This measurement is suggested for any GC peak that has an apparent S/N of less than 5:1. The result of the calculation must appear on the SICP above the GC peak in question.

10.4.5.1. Referring to Table 5, calculate the 17 relative response factors (RRF) for unlabeled target analytes [RRF(n); n=1 to 17] relative to their appropriate isotope dilution analytes (Table 5) and the nine RRFs for the labeled ^{13}C isotope dilution analytes [RRF(m); m=18 to 26] relative to the two internal standards according to the following formulae:

$$RRF(n) = \frac{A_x \times Q_{IDA}}{Q_x \times A_{IDA}} \qquad RRF(m) = \frac{A_{IDA} \times Q_{IS}}{Q_{IDA} \times A_{IS}}$$

Where:

A_x = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 5) for unlabeled PCDDs/PCDFs,

A_{IDA} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 5) for the labeled isotope dilution analytes,

A_{IS} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for the labeled internal standards,

Q_{IDA} = quantity of the isotope dilution analyte injected (pg),

Q_{IS} = quantity of the internal standard injected (pg), and

Q_x = quantity of the unlabeled PCDD/PCDF analyte injected (pg).

The RRF (n) and RRF (m) are dimensionless quantities; the units used to express Q_{IDA}, Q_{IS}, and Qx must be the same.

10.4.5.2. Calculate the RRF(n)s and their respective percent relative standard deviations (%RSD) for the five calibration solutions:

$$\overline{RRF}(n) = (1/5)\sum_{j=1}^{5} RRF_j(n)$$

Where n represents a particular PCDD/PCDF (2,3,7,8-substituted) congener (n = 1 to 17; Table 5), and j is the injection number (or calibration solution number; j = 1 to 5).

10.4.5.3. The relative response factors to be used for the determination of the concentration of total isomers in a homologous series are calculated as follows:

10.4.5.3.1. For congeners that belong to a homologous series containing only one isomer (e.g., OCDD and OCDF) or only one 2,3,7,8-substituted isomer (Table 4; TCDD, PeCDD, HpCDD, and TCDF), the mean RRF used will be the same as the mean RRF determined in Section 10.3.5.2.

NOTE: The calibration solutions do not contain ^{13}C-OCDF as an isotope dilution analyte. This is because a minimum resolving power of 12,000 is required to resolve the [M+6]+ ion of ^{13}C-OCDF from the [M+2]+ ion of OCDD (and [M+4]+ from ^{13}C-OCDF with [M]+ of OCDD). Therefore, the RRF for OCDF is calculated relative to ^{13}C-OCDD.

10.4.5.3.2. For congeners that belong to a homologous series containing more than one 2,3,7,8-substituted isomer (Table 4), the mean RRF used for those homologous series will be the mean of the RRFs calculated for all individual 2,3,7,8-substituted congeners using the equation below:

$$\overline{RRF}(k) = (1/t)\sum_{n=1}^{t} RRF_n$$

Where:

k = 27 to 30, with 27 = PeCDF;

28 = HxCDF; 29 = HxCDD; and 30 = HpCDF,

t = total number of 2,3,7,8-substituted isomers present in the calibration solutions (Table 5) for each homologous series (e.g., two for PeCDF, four for HxCDF, three for HxCDD, two for HpCDF).

NOTE: Presumably, the HRGC/HRMS response factors of different isomers within a homologous series are different. However, this analytical protocol will make the assumption that the HRGC/HRMS responses of all isomers in a homologous series that do not have the 2,3,7,8-substitution patterns are the same as the responses of one or more of the 2,3,7,8-substituted isomer(s) in that homologous series.

10.4.5.4. Relative response factors [RRF(m)] to be used for the determination of the percent recoveries for the nine isotope dilution analytes are calculated as follows:

$$RRF(m) = \frac{A_{IDA}^{m} \times Q_{IS}}{Q_{IDA}^{m} \times A_{IS}}$$

$$\overline{RRF}(m) = (1/5) \sum_{j=1}^{5} RRF_j(m)$$

Where:

m = 18 to 26 (congener type)

j = 1 to 5 (injection number),

A_{IDA}^{m} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given isotope dilution analyte (m = 18 to 26),

A_{IDA} = sum of the integrated ion abundances of the quantitation ions (Tables 6 and 10) for a given isotope dilution analyte (m = 18 to 26),

Q_{IDA} & Q_{IDA}^{m} = quantities of, respectively, the internal standard (rs) and a particular isotope dilution analyte (m) injected (pg),

RRF(m) = relative response factor of a particular isotope dilution analyte (m) relative to an appropriate internal standard, as determined from one injection, and

$\overline{RRF(m)}$ = calculated mean relative response factor of a particular isotope dilution analyte, as determined from the five initial calibration injections (j).

10.5. Criteria for acceptable calibration

The criteria listed below for acceptable calibration must be met before sample analysis is performed.

10.5.1. The percent relative standard deviations for the mean response factors [RRF(n) and RRF(m)] from the 17 unlabeled standards must be ≤ 20 percent, and those for the nine labeled reference compounds must be ≤ 30 percent.

Note: If Method 8290A criteria are required for the project then both the percent standard relative standard deviation for the mean response factors for the 17 unlabeled standards and the nine labeled reference compounds must be ≤ 20 percent.

10.5.2. The signal/noise ratio (S/N) for the GC signals present in every SICP (including the ones for the labeled standards) must be ≥ 10.

10.5.3. The isotopic ratios (Table 9) must be within the specified control limits.

NOTE: If the criterion for acceptable calibration listed in Section 10.4.1 is met, the analyte-specific RRF can then be considered independent of the analyte quantity for the calibration concentration range. The mean RRFs will be used for all calculations until the routine calibration criteria (Section 10.6) are no longer met. At such time, new mean RRFs will be calculated from a new set of injections of the calibration solutions.

10.6. Routine Calibration (continuing calibration check)

Routine calibrations must be performed at the beginning of (following a successful tune and GC column performance check) and after a 12 hour period. The routine calibration initiates the 12 hour clock during which samples may be subsequently analyzed. The last sample in the sequence must be injected within 12 hours of the routine calibration, followed by the analysis of a closing calibration check. An acceptable closing calibration check standard may be used to initiate the next 12 hour analysis sequence when consecutive acquisition sequences occur. The ending mass resolution check shall be performed after the closing calibration check of an analysis acquisition sequence or after the final bracketing standard when consecutive 12 hour acquisition sequences are run.

10.6.1. Inject 1 or 2 µL of the concentration calibration solution HRCC-4 containing 10 pg/µL of tetrachlorinated congeners, 50 pg/µL of penta-, hexa-, and heptachlorinated congeners, 100 pg/µL of octachlorinated congeners, and the respective isotope dilution analyte and internal standards (Table 5). By using the same HRGC/HRMS conditions as used in Sections 6.1.3 through 6.2, determine and document an acceptable calibration as provided in Section 10.6.

10.7. Criteria for Acceptable Routine Calibration

The following criteria must be met before further analysis is performed. If these criteria are not met, corrective action must be taken, including recalibration if needed.

10.7.1. The measured RRFs [RRF(n)] for the unlabeled standards obtained during the opening continuing calibration must be ± 20 percent of the mean values established during the initial calibration (Section 10.3.5.)

10.7.1.1. The bracketing continuing calibration must be ± 20% of the average RRF calculated from the initial calibration.

10.7.1.1.1. If the target compounds in the ending standard are less than or equal to ± 20% of the average RRF from the initial calibration, the RRFs of the initial calibration shall be used to quantitate the unlabeled isomers.

10.7.1.1.2. If the target analytes are greater than ± 20% but less or equal to ±25% and the samples are non-detect, the data is acceptable and this anomaly is documented. If these isomers are greater than ± 20% but less or equal to ±25% and are positive, an average RRF of the initial and ending daily standard is calculated and used to quantitate the concentration of the affected congener, and the anomaly is documented.

10.7.1.1.3. If the percent deviation of unlabeled compounds exceeds ± 25%, a new initial calibration is initiated within 2 hours following the analysis of the samples. Otherwise, reanalyze all sample extracts with positives for the failed target compounds.

10.7.2. The measured RRFs [RRF(m)] for the labeled standards obtained during the opening continuing calibration must be less than or equal to ± 30 percent of the mean values established during the initial calibration (Section 10.1.5).

10.7.2.1. The bracketing continuing calibration must be ± 30% of the average RRF calculated from the initial calibration.

10.7.2.1.1. If the labelled compounds in the ending standard are less than or equal to ±30% of the average RRF from the initial calibration, the RRFs of the initial calibration shall be used to quantitate the labeled isomers.

10.7.2.1.2. If the isotope dilution analyte analytes are greater than ± 30% but less or equal to ±35%, an average RRF of the initial and ending daily standards is calculated and used to quantitate the concentration of the affected congener.

10.7.2.1.3. If the percent deviation of labeled compounds exceeds ± 35%, reanalyze samples if adversely impacted.

10.7.3. The ion-abundance ratios (Table 9) must be within the allowed control limits.

10.7.4. If either criteria in Sections 10.7.1 or 10.7.2 are not met, additional samples may not be analyzed. Sample data collected must be evaluated for usability. Narrate any reported data from the analytical sequence. If the ion-abundance ratio criterion is not satisfied, refer to the note in Section 10.4.3 for resolution.

10.7.5. If the above criteria (Section 10.7) cannot be satisfied, the entire initial calibration process (Section 10.4) must be repeated.

11. PROCEDURE

11.1. Procedural Variations

Procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance memo and approved by a supervisor and QA/QC manager. If contractually required, the client will be notified. The Nonconformance memo will be filed in the project file.

Any deviations from this procedure identified after the work has been completed must be documented as a nonconformance, with a cause and corrective action described. A Nonconformance memo shall be used for this documentation.

11.2. Sample Dilution Procedure – Simple Dilutions

Dilutions from 2X to 50X can be achieved without respiking the final extract. The calculation to determine the final extract concentration is as follows:

(Concentration of the original extract) x (amount of aliquot taken) x (volume of diluted extract) = final concentration of dilution.

Ex: 50X dilution of original 10 g/20 µL sample

(10 g/20 µL) x (2 µL aliquot + 98 µL keeper) = 1 g/100 µL FV

Record the final sample concentration on the extract label.

11.3. Sample Dilution Procedure – Complex Dilutions

Complex dilution requiring respiking of IDA and IS: Dilutions greater than 50x must be done by diluting and respiking the extract with IDA's and IS. This procedure may require serial dilution to be performed. If this procedure is done, then the sample size must be adjusted to reflect the aliquot taken.

Ex. 100X dilution (original sample with 10 g/20 µL final volume)

Take a 2 µL aliquot (1/10 of original sample) and add 18 µL of solvent keeper. Take a 2 µL aliquot of the dilution (1/100 of the original sample), respike with 1 mL IDA and 20 µL IS, reduced to 20 µL FV.

Record the final sample concentration of the extract label.

11.4. Analytical Procedures

11.4.1. Inject a 1 or 2 µL aliquot of the extract into the GC, operated under the conditions previously used (Section 6.2) to produce acceptable results with the performance check solution.

11.4.2. Acquire SIM data according to Section 6.1.3. Use the same acquisition and mass spectrometer operating conditions previously used to determine the relative response factors (Section 10). Ions characteristic for polychlorinated diphenyl ethers are included in the descriptors listed in Table 6. Their presence is used to monitor their interference during the characterization of PCDFs.

12. CALCULATIONS/DATA REDUCTION

12.1. Identification Criteria

For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet all of the following criteria:

12.1.1. Retention Times

12.1.1.1. For 2,3,7,8-substituted congeners, which have an isotopically labeled isotope dilution analyte or internal standard present in the sample extract, the retention time (at maximum peak height) of the sample components (i.e., the two ions used for quantitation purposes listed in Table 6) must be within -1 and +3 seconds of the retention time of the peak for the isotopically labeled isotope dilution analyte or internal standard at m/z corresponding to the first characteristic ion (of the set of two; Table 6) to obtain a positive identification of these nine 2,3,7,8-substituted PCDDs/PCDFs and OCDD.

12.1.1.2. For 2,3,7,8-substituted compounds that do not have an isotopically labeled isotope dilution analyte present in the sample extract, the relative retention time (relative to the appropriate isotope dilution analyte) must fall within 0.005 relative retention time units of the relative retention times measured in the daily routine calibration. Identification of OCDF is based on its retention time relative to ^{13}C-OCDD as determined from the daily routine calibration results.

12.1.1.3. For non-2,3,7,8-substituted compounds (tetra through octa; totaling 119 congeners), the retention time must be within the corresponding homologous retention time windows established by analyzing the column performance check solution.

12.1.1.4. The ion current responses for both ions used for quantitative purposes (e.g., for TCDDs: m/z 319.8965 and 321.8936) must reach a maximum simultaneously (± 2 seconds).

12.1.1.5. The ion current responses for both ions used for the labeled standards (e.g., for ^{13}C-TCDD: m/z 331.9368 and m/z 333.9339) must reach a maximum simultaneously (± 2 seconds).

12.1.2. Ion Abundance Ratios

The integrated ion current for the two ions used for quantitation purposes must have a ratio between the lower and upper limits established for the homologous series to which the peak is assigned. See Table 9.

12.1.3. Signal-To-Noise Ratio

All ion current intensities must be >2.5 times noise level for positive identification of the PCDD/PCDF compound or a group of coeluting isomers. Figure 4 describes the procedure to be followed for the determination of the S/N.

12.1.4. Polychlorinated Diphenyl Ether Interferences

In addition to the above criteria, the identification of a GC peak as a PCDF can only be made if no signal having a S/N >2.5 is detected, at the same retention time (± 2 seconds), in the corresponding polychlorinated diphenyl ether (PCDPE, Table 6) channel.

12.2. For gas chromatographic peaks that have met the criteria outlined above, calculate the concentration of the PCDD or PCDF compounds using the formula:

$$C_x = \frac{A_x \times Q_{IDA}}{A_{IDA} \times W \times RRF(n)}$$

Where:

C_x = concentration of unlabeled PCDD/PCDF congeners (or group of coeluting isomers within an homologous series) usually in pg/g or pg/L,

Ax = sum of the integrated ion abundances of the quantitation ions (Table 6) for the unlabeled PCDD/PCDFs,

A_{IDA} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled isotope dilution analytes,

Q_{IDA} = quantity, in pg, of the isotope dilution analyte added to the sample before extraction,

W = sample size in g (if solid) or L (if liquid).

$RRF(n)$ = Calculated mean relative response factor for the analyte [RRF(n) with n = 1 to 17; Section 10.3.5].

If the analyte is identified as one of the 2,3,7,8-substituted PCDDs or PCDFs, RRF(n) is the value calculated using the equation in Section 10.3.5.1. However, if it is a non-2,3,7,8-substituted congener, the RRF(k) value is the one calculated using the equation in Section 10.3.5.3.2 [RRF(k) with k = 27 to 30].

12.3. Calculate the percent recovery of the nine isotope dilution analytes measured in the sample extract, using the formula:

$$\text{Isotope Dilution Analytes Percent Recovery} = \frac{A_{IDA} \times Q_{IS}}{Q_{IDA} \times A_{IS} \times RRF(m)} \times 100$$

Where:

A_{IDA} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled isotope dilution analytes,

A_{IS} = sum of the integrated ion abundances of the quantitation ions (Table 6) for the labeled internal standard; the selection of the internal standard depends on the type of congeners (see Table 5, footnotes),

Q_{IDA} = Quantity, in pg, of the isotope dilution analyte added to the sample before extraction,

Q_{IS} = Quantity, in pg, of the internal standard added to the cleaned-up sample residue before HRGC/HRMS analysis, and

$RRF(m)$ = calculated mean relative response factor for the labeled isotope dilution analyte relative to the appropriate (see Table 5, footnotes) internal standard. This represents the mean obtained in Section 10.3.5.4 [RRF(m) with m = 18 to 26].

12.4. If the concentration in the final extract of any of the fifteen 2,3,7,8-substituted PCDD/PCDF compounds (Table 3) exceeds the upper method calibration limit (MCL) for that compound listed in Table 1, the linear range of response versus concentration may have been exceeded. In such cases, the following corrective actions will be undertaken:

12.4.1. If the signal for the analyte has saturated the detector, a single dilution and reanalysis of the extract will be made in an attempt to bring the signal within the range of the detector. If the measured concentration of the analyte is still above the MCL, the reported concentration for the analyte will be qualified appropriately. Some programs, such as DOD QSM, require all compounds to be within the linear calibration range in which a serial dilution must be performed to achieve acceptable quantitation.

12.4.2. If the signal for the analyte is above the MCL but does not saturate the detector, the concentration will be reported and qualified appropriately. Some programs, such as DOD QSM, require all compounds to be within the linear calibration range in which a serial dilution must be performed to achieve acceptable quantitation.

12.5. In either case, **with the approval of the client**, the sample may be re-extracted and/or re-analyzed with one or more of the following adjustments made to the analytical procedure in order to provide a concentration which meets client-specific data quality objectives.

12.5.1. Extraction and analysis of a one tenth aliquot. This is appropriate if it will provide analyte concentration within the MCL and a representative sample aliquot.

12.5.2. Extraction of an aliquot large enough to be representative with an increased concentration of isotope dilution analyte and surrogate spike components added prior to the extraction. The extract is then diluted either prior to or after the cleanup procedures.

12.5.3. Dilution of the original extract. Isotope dilution analyte components are re-spiked at an appropriate level prior to analysis. In this case, the isotope dilution analyte recoveries are taken from the original analysis.

12.6. For the other congeners (including OCDD and OCDF), however, report the measured concentration and indicate that the value exceeds the upper calibration standard.

12.7. The total concentration for each homologous series of PCDD and PCDF is calculated by summing up the concentrations of all positively identified isomers of each homologous series. Therefore, the total should also include the 2,3,7,8-substituted congeners. The total number of GC signals included in the homologous total concentration value may be specified in the report.

12.8. Sample-Specific Estimated Detection Limit

The sample-specific estimated detection limit (EDL) or estimated quantiation limit (EQL, 8290A) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. An EDL/EQL is calculated for each 2,3,7,8-substituted congener that is not identified, regardless of whether or not other non-2,3,7,8-substituted isomers are present. Two methods of calculation can be used, as follows, depending on the type of response produced during the analysis of a particular sample.

12.8.1. Samples giving a response for both quantitation ions (Tables 6 and 9) that is less than 2.5 times the background level.

Use the expression for EDL/EQL (specific 2,3,7,8-substituted PCDD/PCDF) below to calculate an EDL/EQL for each absent 2,3,7,8-substituted PCDD/PCDF (i.e., S/N <2.5). The background level is determined by measuring the range of the noise (peak to peak) for the two quantitation ions (Table 6) of a particular 2,3,7,8-substituted isomer within an homologous series, in the region of the SICP trace corresponding to the elution of the isotope dilution analyte (if the congener possesses an isotope dilution analyte) or in the region of the SICP where the congener is expected to elute by comparison with the routine calibration data (for those congeners that do not have a ^{13}C-labeled standard), multiplying that noise height by 2.5, and relating the product to an estimated concentration that would produce that product height.

NOTE: The quantitation ions for both the unlabeled PCDDs/PCDFs and their isotope dilution analyte must be consistently paired (using either both lighter mass ions or both heavier mass ions).

Use the formula:

$$EDL_{Specific\,2,3,7,8-subst.\,PCDD/PCDF} = \frac{2.5 \times H_x \times Q_{IDA}}{H_{IDA} \times W \times RRF(n)}$$

Where:

EDL = estimated detection limit for homologous 2,3,7,8-substituted PCDDs/PCDFs. (also EQL for Method 8290A)

H_x = height of the average noise for one of the quantitation ions (Table 6) for the unlabeled PCDDs/PCDFs.

H_{IDA} = height of one of the quantitation ions (Table 6) for the labeled isotope dilution analytes.

W, RRF (n), and Q_{IDA} retain the same meanings as defined in Section 12.2

12.8.2. Samples characterized by a response above the background level with a S/N of at least 2.5 for at least one of the quantitation ions (Tables 6 and 9).

When the response of a signal having the same retention times as a 2,3,7,8-substituted congener has a S/N in excess of 2.5 and does not meet any of the other qualitative identification criteria listed in Section 12.1, calculate the "Estimated Maximum Possible Concentration" (EMPC) according to the expression shown in Section 12.1, except that Ax in Section 12.1 should represent the sum of the area under the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio. Alternatively, an EDLEQL can be calculated using the above formula and the height of one of the ions as appropriate.

12.9. The relative percent difference (RPD) is calculated as follows:

$$RPD = \frac{|S_1 - S_2|}{(S_1 + S_2)/2} \times 100$$

S_1 and S_2 represent sample and duplicate sample results.

12.10. The 2,3,7,8-TCDD toxic equivalents (TEQ) of PCDDs and PCDFs present in the sample are calculated at the data user's request. This method assigns a 2,3,7,8-TCDD toxicity equivalency factor (TEF) to each of the seventeen 2,3,7,8-substituted PCDDs and PCDFs (Table 10). The 2,3,7,8-TCDD equivalent of the PCDDs and PCDFs present in the sample is calculated by summing the TEF times their concentration for each of the compounds or groups of compounds listed in Table 10.

12.11. Two-GC Column TEF Determination

12.11.1. The concentration of 2,3,7,8-TCDD (see note below), is calculated from the analysis of the sample extract on the 60m DB-5 fused silica capillary column. The chromatographic separation of this isomer must be $\leq$ 25% valley.

12.11.2. For samples that have a positive result for 2,3,7,8-TCDF on the DB-5 column, the extract is reanalyzed on a 30m DB-225 fused silica column. The GC/MS conditions are altered so that only the first descriptor (Table 6) is used. The reported concentration for 2,3,7,8-TCDF is then the result above the lower calibration limit is calculated from the DB-225 analysis. The chromatographic separation between 2,3,7,8-TCDF and any other unlabeled TCDF isomers must be < 25% valley using the column performance check solution for the DB-225 column. Concentration calculations are performed as in Section 12.1 through 12.6.

12.11.3. A DB-225 column can be used in the quantitative analysis of 2,3,7,8-TCDF and 2,3,7,8-TCDD analytes. Since the DB-225 cannot resolve 2,3,7,8-TCDD any positively identified 2,3,7,8-TCDD which exceeds the reporting limit shall be confirmed on a DB-5 column.

12.11.4. For a gas chromatographic peak to be identified as a 2,3,7,8-substituted PCDD/PCDF congener, it must meet the ion abundance (Section 11.5.4) and signal-to-noise ratio criteria. In addition, the retention time identification criterion described in Section 11.5.4 applies here for congeners for which a carbon-labeled analog is available in the sample extract. However, the relative retention time (RRT) of the 2,3,7,8-substituted congeners for which no carbon-labeled analogs are available must fall within 0.006 units of the carbon-labeled standard RRT. Experimentally, this is accomplished by using the attributions described in Table 11 and the results from the routine

calibration run on the DB-5 column.

13. METHOD PERFORMANCE

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

It must be documented that all applicable system performance criteria specified were met before analysis of any sample is performed. Table 7 provides recommended GC conditions that can be used to satisfy the required criteria. A GC column performance check is only required at the beginning of each 12-hour period during which samples are analyzed.

13.5. GC Column Performance

13.5.1. Inject 1 or 2 µL of the column performance check solution and acquire selected ion monitoring (SIM) data as described in Section 6.1.3 within a total cycle time of < 1 second.

13.5.2. The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other TCDD isomers must be resolved with a valley of ≤ 25

percent (Figure 2),

Where:

$$\text{Valley Percent} = (x/y) \times 100$$

x = measured as in Figure 2 from the 2,3,7,8-closest TCDD eluting isomer,

y = the peak height of 2,3,7,8-TCDD

13.5.3. It is the responsibility of the laboratory to verify the conditions suitable for the appropriate resolution of 2,3,7,8-TCDD from all other TCDD isomers. The GC column performance check solution also contains the known first and last PCDD/PCDF eluters under the conditions specified in this protocol. Their retention times are used for qualitative and quantitative purposes. The peak for 2,3,7,8-TCDD must be labeled on the chromatograms. The chromatograms showing the first and last eluters of a homologous series must be included.

13.5.4. The retention times for the switching of SIM ions characteristic of one homologous series to the next higher homologous series must be indicated in the SICP. Accurate switching at the appropriate times is absolutely necessary for accurate monitoring of these compounds.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Autovials containing assorted solvents and extracts. As the autovials are removed from the instrument after analysis, they are collected in archive boxes and retained pending additional instructions. When no longer needed, the archive boxes are moved to the waste disposal area for disposal as PCB waste.

16. REFERENCES/CROSS REFERENCES

16.1. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 8290 Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry September 1994.

16.2. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update IV. Method 8290A Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry February 2007.

16.3. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 0023A, Sampling Method for Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans Emissions from Stationary Sources. December 1996.

16.4. Compendium Method TO-9A “Determination of Polychlorinated, Polybrominated, and Brominated, Cholorinated Dibenxo-p-dioxins and Dibenzofurans in Ambient Air”, EPA compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, second edition, January 1997.

16.5. Protocol for the Analysis of 2,3,7,8-TCDD by HRGC/HRMS". J. S. Stanley and T. M. Sack, EPA 600/4-86-004.

16.6. “Safety in Academic Chemistry Laboratories", American Chemical Society Publication, Committee on Chemical Safety (3rd Edition, 1979.)

16.7. “Carcinogens - Working with Carcinogens". Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control. National Institute for Occupational Safety and Health. Publication No. 77-206, August 1977.

16.8. "OSHA Safety and Health Standards, General Industry", (29 CFR 1910) Occupational Safety and Health Administration, OSHA 2206 (revised January 1976).

17. METHOD MODIFICATIONS

17.1. Modifications from EPA 8290 and EPA 8290A

17.1.1. The methods specify that 2 µL injections are used throughout the analysis. If an instrument demonstrates adequate sensitivity and chromatographic resolution, then the analyst may use 1 µL injections for all performance checks, standards, QC samples, and samples.

17.1.2. In Section 2.7 of Method 8290 and 8290A, a retention time window of 0.005 RT units is used to tentatively identify unlabeled PCDD/PCDFs for which there are no corresponding labeled isotope dilution analytes. All available labeled isotope dilution analytes are used; therefore, a retention time window

of -1 to +3 seconds is used to identify all compounds. See Section 7.8.4.1 of Method 8290 and 7.9 of Method 8290A.

17.1.3. Tetradecane instead of nonane is used as the final solvent to increase the stability of extracts and standards. Tetradecane is less volatile than nonane. Loss of analyte as a result of solvent incompatibility is monitored through recovery checks and calibration acceptance criteria.

17.2. Modifications from TO-9A method

17.2.1. The ^{37}Cl-2,3,7,8-TCDD surrogate is present at varying levels in the calibration curve (0.5-200 pg/ µL).

17.2.2. The laboratory uses 2 labeled internal standards for the quantitation of labeled isotope dilution analytes.

17.2.3. The final volume is adjusted to 20 µL in tetradecane.

17.2.4. Calibration and quantitation are performed in accordance to this SOP.

18. ATTACHMENTS

18.1. Table 1 - Types of Matrices

18.2. Table 2 - Composition of the Sample Fortification and Internal Standard Solutions.

18.3. Table 3 - The Fifteen 2,3,7,8-Substituted PCDD and PCDF Congeners

18.4. Table 4 - Isomers of Chlorinated Dioxins and Furans

18.5. Table 5 - Concentrations of Calibration Solutions

18.6. Table 6 - Ions Monitored for PCDDs/PCDFs

18.7. Table 7 - Recommended GC Operating Conditions

18.8. Table 8 - Congeners in the GC Performance Evaluation Solution (DB-5)

18.9. Table 9 - Theoretical Ion Abundance Ratios and Control Limits

18.10. Table 10 - 2,3,7,8-TCDD Equivalent Factors

18.11. Table 11 - TEF: Analyte Relative Retention Time Reference Attributes

18.12. Figure 1 - Compound Structure

18.13. Figure 2 - GC Performance Check Chromatogram on the DB-5 Column

18.14. Figure 3 - PFK Peak Profile

18.15. Figure 4 - Manual Determination of Signal-to-Noise

18.16. Appendix A - Periodic Wipe Test Performance

19. REVISION HISTORY

19.1. WS-ID-0005, Revision 7.5, Effective 04/19/2013

19.1.1. Replaced all instances of ‘internal standard’ with isotope dilution analyte’ and all instances of ‘recovery standard’ with ‘internal standard’ to conform with TALS naming guidelines.

19.1.2. Editorial revisions.

19.2. WS-ID-0005, Revision 7.4, Effective 01/14/2011.

19.2.1. Editorial revisions.

19.3. WS-ID-0005, Revision 7.3, Effective 12/30/2009

19.3.1. Editorial revisions.

19.4. WS-ID-0005, Revision 7.2, Effective 11/02/2009

19.4.1. Section 6.1: Inserted “Preventive and routine maintenance is described in the ‘Schedule of Routine Maintenance’ in the QAM.”

19.4.2. Section 12.1.2: Removed the word "presumptive" and inserted "above the lower calibration limit" after the word result.

TABLE 1

Types of Matrices, Sample Sizes and 2,3,7,8-TCDD-Based Method Calibration Limits (Parts per Trillion)

	Water	Soil Sediment Paper Pulp	Fly Ash	Human/ Fish Tissue	Adipose Tissue	Sludges, Fuel Oil	Still-Bottom	Ambient or Source Samples
Lower MCL(a)	0.01	1.0	2.0	1.0	2.0	10	20	40
Upper MCL(a)	4.0	400	400	400	400	2000	4000	8000
Weight (g)	1000	10	10	10	10	2.0	1.0	1 sample
IDA Spiking Levels (ng)	2.0	2.0	2.0	2.0	2.0	2.0	2.0	4.0
Final Extract Volume (µL)	20	20	20	20	20	20	20	20

(a) For other congeners, multiply the values by 1 for TCDF, by 5 for PeCDD/PeCDF/HxCDD/HxCDF/HpCDD/HpCDF, and by 10 for OCDD/OCDF.

TABLE 2

Composition of the Sample Fortification and Internal Standard Solutions

Analyte	Sample Fortification Solution Concentration pg/µL; Solvent: Isooctane	Internal Standard Solution Concentration pg/µL; Solvent: Tetradecane
^{13}C-2,3,7,8-TCDD	2[a], 100[c]	--
^{13}C -2,3,7,8-TCDF	2[a], 100[c]	--
^{13}C -1,2,3,4-TCDD	--	100
^{13}C -1,2,3,7,8-PeCDD	2[a], 100[c]	--
^{13}C -1,2,3,7,8-PeCDF	2[a], 100[c]	--
^{13}C -1,2,3,6,7,8-HxCDD	2[a], 100[c]	--
^{13}C -1,2,3,4,7,8-HxCDF[d]	2[a], 100[c]	--
^{13}C -1,2,3,7,8,9-HxCDD	--	100
^{37}Cl-2,3,7,8-TCDD[b][c]	0.8[b], 100[c]	
	100[c]	
^{13}C -2,3,4,7,8-PeCDF[c]	100[c]	
^{13}C -1,2,3,6,7,8-HxCDF[c][d]	100[c]	
^{13}C -1,2,3,4,7,8-HxCDD[c]	100[c]	
^{13}C -1,2,3,4,7,8,9-HpCDD[c]	100[c]	
^{13}C -1,2,3,4,6,7,8-HpCDD	2[a], 100[c]	--
^{13}C -1,2,3,4,6,7,8-HpCDF	2[a], 100[c]	--
^{13}C -OCDD	4[a], 200[c]	--

(a) Standard 8290, 8290A, Method 23, Method 0023A, TO9 and TO9A Sample Fortification Solution concentrations
(b) Method TO9 and TO9A surrogate concentrations
(c) Method 23 and Method 0023A surrogate concentrations
(d) ^{13}C-1,2,3,6,7,8-HxCDF is used as a Sample Fortification Solution and ^{13}C -1,2,3,4,7,8-HxCDF is used as a surrogate solution in Method 0023A

TABLE 3

The Seventeen 2,3,7,8-Substituted PCDD and PCDF Congeners

PCDD	PCDF
2,3,7,8-TCDD(*)	2,3,7,8-TCDF(*)
1,2,3,7,8-PeCDD(*)	1,2,3,7,8-PeCDD(*)
1,2,3,6,7,8-HxCDD(*)	2,3,4,7,8-PeCDF
1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDD(+)	1,2,3,7,8,9-HxCDF
1,2,3,4,6,7,8-HpCDD(*)	1,2,3,4,7,8-HxCDF(*)
1,2,3,4,5,6,7,8-OCDD(*)	2,3,4,6,7,8-HxCDF
	1,2,3,4,6,7,8-HpCDF(*)
	1,2,3,4,7,8,9-HpCDF
	1,2,3,4,5,6,7,8-OCDF

(*)The ^{13}C -labeled analog is used as an isotope dilution analyte.
(+)The ^{13}C -labeled analog is used as a internal standard.

TABLE 4

Isomers of Chlorinated Dioxins and Furans as a Function of the Number of Chlorine Atoms

# of Chlorine Atoms	# of Dioxin Isomers	# of 2,3,7,8 Isomers	# of Furan Isomers	# of 2,3,7,8 Isomers
1	2	---	4	---
2	10	---	16	---
3	14	---	28	---
4	22	1	38	1
5	14	1	28	2
6	10	3	16	4
7	2	1	4	2
8	1	1	1	1
Total	**75**	**7**	**135**	**10**

TABLE 5

High Resolution Concentration Calibration Solutions

	Compound	Concentration (ng/mL)				
RRF (n)(m)		**CS2**	**CS3**	**CS4 (ICV(6))**	**CS5**	**CS6**
	Native CDDs and CDFs					
1	2,3,7,8-TCDD	0.5	2	10	40	200
2	2,3,7,8-TCDF	0.5	2	10	40	200
3	1,2,3,7,8-PeCDD	2.5	10	50	200	1000
4	1,2,3,7,8-PeCDF	2.5	10	50	200	1000
5	2,3,4,7,8-PeCDF	2.5	10	50	200	1000
6	1,2,3,4,7,8-HxCDD	2.5	10	50	200	1000
7	1,2,3,6,7,8-HxCDD	2.5	10	50	200	1000
8	1,2,3,7,8,9-HxCDD	2.5	10	50	200	1000
9	1,2,3,4,7,8-HxCDF	2.5	10	50	200	1000
10	1,2,3,6,7,8-HxCDF	2.5	10	50	200	1000
11	1,2,3,7,8,9-HxCDF	2.5	10	50	200	1000
12	2,3,4,6,7,8-HxCDF	2.5	10	50	200	1000
13	1,2,3,4,6,7,8-HpCDD	2.5	10	50	200	1000
14	1,2,3,4,6,7,8-HpCDF	2.5	10	50	200	1000
15	1,2,3,4,7,8,9-HpCDF	2.5	10	50	200	1000
16	OCDD	5.0	20	100	400	2000
17	OCDF	5.0	20	100	400	2000
	Labeled CDDs and CDFs					
18	$^{13}C_{12}$-2,3,7,8-TCDD	100	100	100	100	100
19	$^{13}C_{12}$-2,3,7,8-TCDF	100	100	100	100	100
20	$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	100	100	100	100
21	$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	100	100	100	100
	$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	100	100	100	100
22	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	100	100	100	100
23	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	100	100	100	100
	$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	100	100	100	100
24	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	100	100	100	100
25	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,4,7,8,9-	100	100	100	100	100

	Compound	Concentration (ng/mL)				
RRF (n)(m)		**CS2**	**CS3**	**CS4 (ICV(6))**	**CS5**	**CS6**
	HpCDF					
26	$^{13}C_{12}$-OCDD	200	200	200	200	200
	Cleanup Standard/ FS					
	$^{37}Cl_4$-2,3,7,8-TCDD	0.5	2	10	40	200
	Internal Standards					
	$^{13}C_{12}$-1,2,3,4-TCDD	100	100	100	100	100
	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	100	100	100	100	100

TABLE 6*
Elemental Compositions and Exact Masses of the Ions
Monitored by HR/MS for PCDD's and PCDF's

Descriptor	Exact m/z [1]	m/z Type	Elemental Composition	Substance [2]
1	292.9825	QC	C_7F_{11}	PFK
	303.9016	M	$C_{12}H_4{}^{35}Cl_4O$	TCDF
	305.8987	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF
	315.9419	M	$^{13}C_{12}H_4{}^{35}Cl_4O$	TCDF [3]
	317.9389	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF [3]
	319.8965	M	$C_{12}H_4{}^{35}Cl_4O_2$	TCDD
	321.8936	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD
	327.8847	M	$C_{12}H_4{}^{37}Cl_4O_2$	TCDD [4]
	330.9792	Lock	C_7F_{13}	PFK
	331.9368	M	$^{13}C_{12}H_4{}^{35}Cl_4O_2$	TCDD [3]
	333.9339	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD [3]
	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	341.8567	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}ClO$	PeCDF
	375.8364	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}ClO$	HxCDPE
	409.7974	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}ClO$	HpCDPE
2	330.9792	QC	C_7F_{13}	PFK
	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	341.8567	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF
	342.9792	Lock	C_8F_{12}	PFK
	351.9000	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	353.8970	M+4	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF [3]
	354.9792	Lock	C_9F_{13}	PFK
	355.8546	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD
	357.8516	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD
	366.9793	QC	C_9F_{13}	PFK
	367.8949	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD [3]
	369.8919	M+4	$^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD [3]
	409.7974	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}ClO$	HpCDPE
3	373.8208	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF
	375.8178	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$	HxCDF
	380.9760	Lock	C_8F_{15}	PFK
	383.8639	M	$^{13}C_{12}H_2{}^{35}Cl_6O$	HxCDF [3]
	385.8610	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF [3]
	389.8157	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD
	391.8127	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD
	392.9760	Lock	C_9F_{15}	PFK
	401.8559	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD [3]
	403.8529	M+4	$^{13}C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD [3]
	430.9728	QC	C_9F_{17}	PFK
	445.7550	M+4	$C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$	OCDPE
4	392.9760	QC	C_9F_{15}	PFK
	407.7818	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF
	409.7789	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O$	HpCDF

Descriptor	Exact m/z [1]	m/z Type	Elemental Composition	Substance [2]
	417.8253	M	$^{13}C_{12}H^{35}Cl_7O$	HpCDF [3]
	419.8220	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF [3]
	423.7766	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD
	425.7737	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD
	430.9729	Lock	C_9F_{17}	PFK
	435.8169	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD [3]
	437.8140	M+4	$^{13}C_{12}H^{35}Cl_5{}^{37}CL_2O_2$	HpCDD [3]
	479.7165	M+4	$C_{12}H^{35}Cl_7{}^{37}Cl_2O$	NCDPE
5	392.9760	QC	C_9F_{15}	PFK
	441.7428	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO$	OCDF
	442.9728	Lock	$C_{10}F_{17}$	PFK
	443.7399	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O$	OCDF
	457.7377	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD
	459.7348	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD
	469.7779	M+2	$^{13}C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD [3]
	471.7750	M+4	$^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD [3]
	479.7165	M+4	$C_{12}Cl_8{}^{37}Cl_2O$	NCDPE
	513.6775	M+4	$^{13}C_{12}{}^{35}Cl_8{}^{37}Cl_2O$	DCDPE

---

[a] The following nuclidic masses were used:

H =	1.007825	O =	15.994915
C =	12.000000	^{35}Cl =	34.968853
^{13}C =	13.003355	^{37}Cl =	36.965903
F =	18.9984		

S = Isotope dilution analyte/internal standard

*The homologous groups for functions 1-3 do not use the same lockmass as described in Table 6. They use masses 316.9824, 366.9792, and 380.9760, respectively.

TABLE 7

Recommended GC Operating Conditions

The GC Operating Conditions (Temperatures (°C), and Times (minutes)) Are as Follows:

Injector Temperature: 280°C
Interface Temperature: 280°C
Initial Temperature and Time: 190°C / 1 Minute

Temperature Program: 190°C, increasing at a rate of 4°C per minute up to 240°C, and maintaining at this temperature until the last tetra of the tetra- group has eluted from the column. (The total time required for this is approximately 25 minutes, depending on the length of the column). The maintained temperature of 240°C is then increased to 320°C at the rate of 20°C per minute and held at this level until the last compound (octa-group) has eluted from the column.

TABLE 8

PCDD and PCDF Congeners Present in the GC Performance Evaluation Solution and Used for Defining the Homologous GC Retention Time Windows on a 60-M DB-5 Column[(b)]

# of Chlorine Atoms	PCDD Positional Isomer		PCDF Positional Isomer	
	Early Eluter	Late Eluter	Early Eluter	Late Eluter
4[(a)]	1,3,6,8	1,2,8,9	1,3,6,8	1,2,8,9
5	1,2,4,6,8/1,2,4,7,9	1,2,3,8,9	1,3,4,6,8	1,2,3,8,9
6	1,2,3,4,6,8	1,2,3,4,6,7	1,2,3,4,6,8	1,2,3,4,8,9
7	1,2,3,4,6,7,8	1,2,3,4,6,7,9	1,2,3,4,6,7,8	1,2,3,4,6,7,9
8	1,2,3,4,6,7,8,9		1,2,3,4,6,7,8,9	

[(a)] In addition to these two PCDD isomers, the 1,2,3,4-, 1,2,3,7-, 1,2,3,8-, 2,3,7,8-, $^{13}C_{12}$-2,3,7,8-, and 1,2,3,9-TCDD isomers must also be present.

(b) The PCDF Congeners present in GC the Performance Evaluation Solution for the 30 m DB-225 column include:

- 1,2,3,9-TCDF
- 2,3,7,8-TCDF
- 2,3,4,7-TCDF
- $^{13}C_{12}$-2,3,7,8-TCDF

Column performance criteria is met when the percent valleys between the 2,3,7,8-TCDF analyte and the closest eluting isomers are $\leq$ 25%.

TABLE 9

Theoretical Ion Abundance Ratios and Their Control Limits for PCDDs and PCDFs

# of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits	
			Lower	Upper
4	M / M+2	0.77	0.65	0.89
5	M+2 / M+4	1.55	1.32	1.78
6	M+2 / M+4	1.24	1.05	1.43
6[(a)]	M / M+2	0.51	0.43	0.59
7[(b)]	M / M+2	0.44	0.37	0.51
7	M+2 / M+4	1.04	0.88	1.20
8	M+2 / M+4	0.89	0.76	1.02

(a) Used only for ^{13}C-HxCDF (IS) (b) Used only for ^{13}C-HpCDF (IS)

TABLE 10

2,3,7,8-TCDD Equivalent Factors (TEFs) for the Polychlorinated Dibenzodioxins and Dibenzofurans

Number	Compound(s)	TEF
1	2,3,7,8-TCDD	1.00
2	1,2,3,7,8-PeCDD	0.50
3	1,2,3,6,7,8-HxCDD	0.10
4	1,2,3,7,8,9-HxCDD	0.10
5	1,2,3,4,7,8-HxCDD	0.10
6	1,2,3,4,6,7,8-HpCDD	0.01
7	OCDD	0.001
8	2,3,6,7-TCDF	0.1
9	1,2,3,7,8-PeCDF	0.05
10	2,3,4,7,8PeCDF	0.5
11	1,2,3,6,7,8-HxCDF	0.1
12	1,2,3,7,8,9-HxCDF	0.1
13	1,2,3,4,7,8-HxCDF	0.1
14	2,3,4,6,7,8-HxCDF	0.1
15	1,2,3,4,6,7,8-HpCDF	0.01
16	1,2,3,4,7,8,9-HpCDF	0.01
17	OCDF	0.001

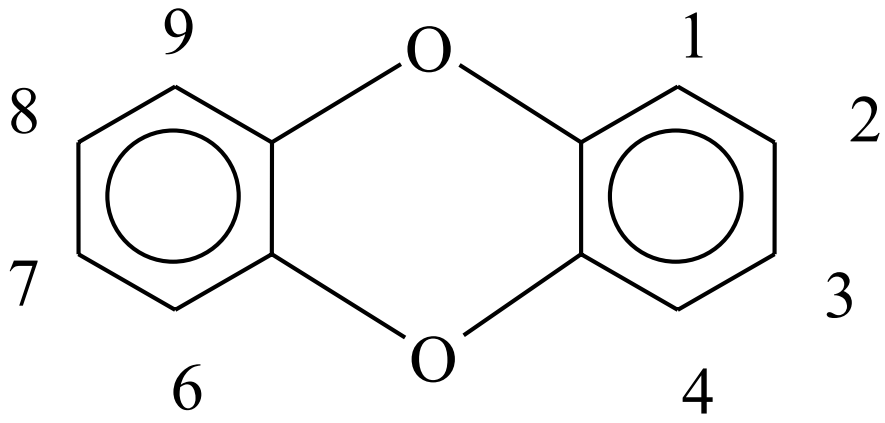
TABLE 11

Toxicity Equivalency Factor:
Analyte Relative Retention Time Reference Attributes

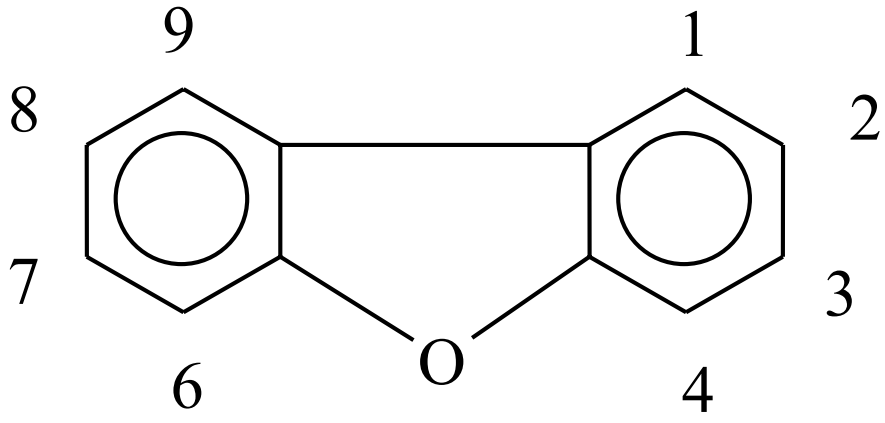
Analyte	Analyte RRT Reference (a)
1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF
1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF
2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,4,7,8-HxCDF

(a) The retention time of 2,3,4,7,8-PeCDF on the DB-5 column is measured relative to $^{13}C_{12}$-1,3,7,8-PeCDF and the retention time of 1,2,3,4,7,8,9-HpCDF relative to $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF

FIGURE 1
Structure of Dibenzodioxin and Dibenzofuran

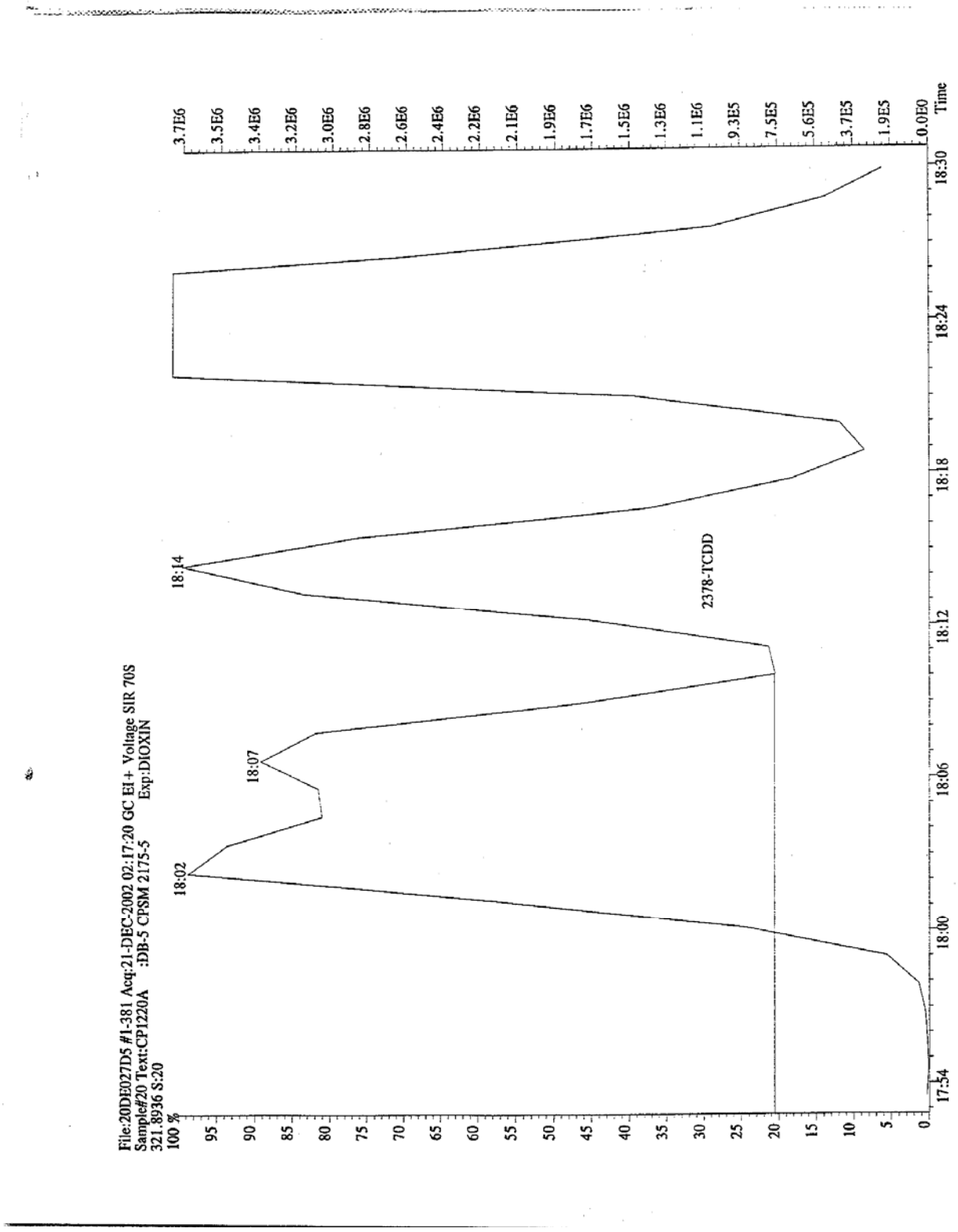


Dibenzodioxin



Dibenzofuran

FIGURE 2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Sample#20 Text:CP1220A :DB-5 CPSM 2175-5 Exp:DIOXIN
321.8936 S:20
100 %
18:02
18:07
18:14
2378-TCDD
Time
17:54
18:00
18:06
18:12
18:18
18:24
18:30
3.7E6
3.5E6
3.4E6
3.2E6
3.0E6
2.8E6
2.6E6
2.4E6
2.2E6
2.1E6
1.9E6
1.7E6
1.5E6
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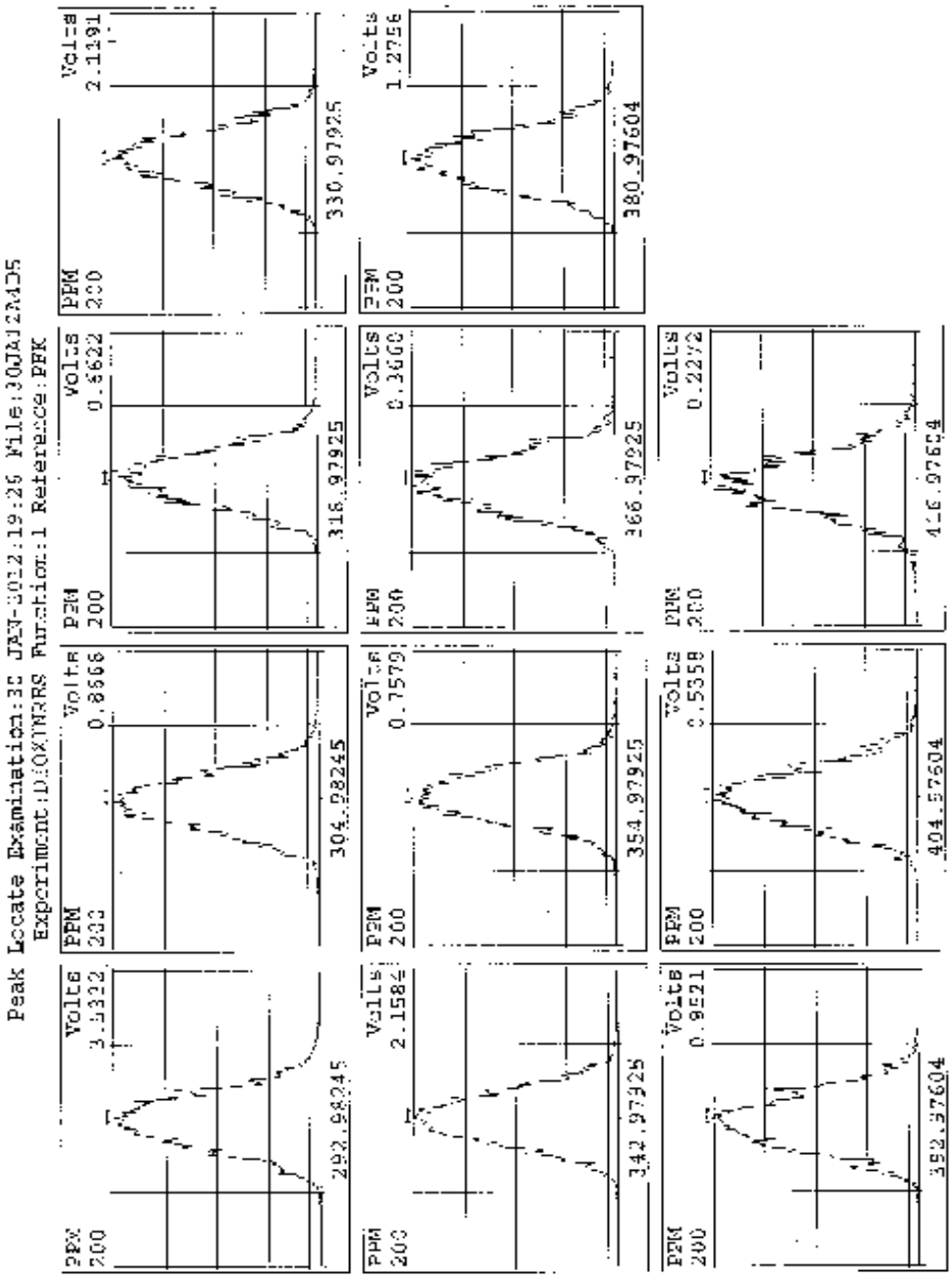
5.6E5
3.7E5
1.9E5
0.0E0

Figure 3


Peak Locate Examination:3S JAN-2012 19:25 File:30JAN2A35
Experiment:U:OXINSRS Function:1 Reference:PFK
PPM
200
Volts
3.3322
292.98245
PPM
200
Volts
0.8668
304.98245
PPM
200
Volts
0.9622
316.97925
PPM
200
Volts
2.1191
330.97925
PPM
200
Volts
2.1582
342.97925
PPM
200
Volts
0.7579
354.97925
PPM
200
Volts
0.3660
366.97925
PPM
200
Volts
1.2756
380.97604
PPM
200
Volts
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PPM
200
Volts
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PPM
200
Volts
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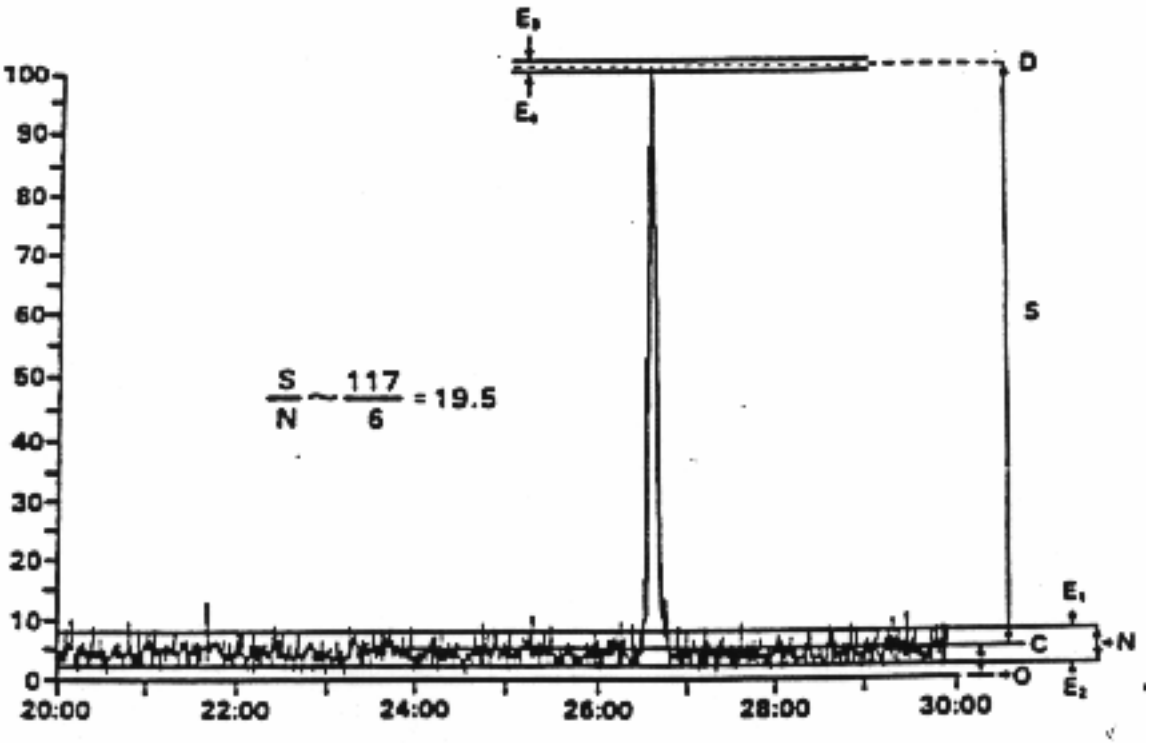
416.97604

FIGURE 4



Manual determination of S/N.

The peak height (S) is measured between the mean noise (lines C and D). These mean signal values are obtained by tracing the line between the baseline average noise extremes, E1 and E2, and between the apex average noise extremes, E3 and E4, at the apex of the signal.

NOTE: It is imperative that the instrument interface amplifier electronic zero offset be set high enough so that negative going baseline noise is recorded.

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Revision 0
November 1992

APPENDIX A

This procedure is designed for the periodic evaluation of potential contamination by 2,3,7,8-substituted PCDD/PCDF congeners of the working areas inside the laboratory.

PERFORMING WIPE TEST

Perform the wipe tests on surface areas of two inches by one foot with laboratory wipers saturated with distilled-in-glass acetone or appropriate solvent using a pair of clean stainless steel forceps. Use one wiper for each of the designated areas. Combine the wipers to one composite sample in an extraction jar containing 200 mL distilled-in-glass hexane. Place an equal number of unused wipers in 200 mL hexane and use this as a control.

SAMPLE PREPARATION

Close the jar containing the wipes and 200 mL hexane and extract for 20 minutes using a wrist-action shaker. Use an appropriate means to reduce the volume to approximately 1.0 mL. Put through an alumina column to clean up potential interfering compounds. Add appropriate amount of internal standard.

EXTRACT ANALYSIS

Concentrate the contents of the vial to a final volume of 20 µL (either in a minivial or in a capillary tube). Inject 2 µL of each extract (wipe and control) onto a capillary column and analyze for 2,3,7,8-substituted PCDDs/PCDFs as specified in the analytical method Section 11 (this exhibit). Perform calculations according to Section 12 (this exhibit).

REPORTING FORMAT

Report the presence of 2,3,7,8-substituted PCDDs and PCDFs as a quantity (pg or ng) per wipe test experiment (WTE). Under the conditions outlined in this analytical protocol, a lower limit of calibration of 25 pg/WTE is expected for 2,3,7,8-TCDD. A positive response for the blank (control) is defined as a signal in the TCDD retention time window at any of the masses monitored which is equivalent to or above 8 pg of 2,3,7,8-TCDD per WTE. For other congeners, use the multiplication factors listed in Table 1, footnote (a) (e.g., for OCDD, the lower MCL is 25 x 5 = 125 pg/WTE and the positive response for the blank would be 8 x 5 = 40 pg). Also, report the recoveries of the isotope dilution analytes during the simplified cleanup procedure.

FREQUENCY OF WIPE TESTS

Wipe tests should be performed when there is evidence of contamination in the method blanks.

CORRECTIVE ACTION

An upper limit of 25 pg per TCDD isomer and per wipe test experiment is allowed. (Use multiplication factors listed in footnote (a) from Table 1 for other congeners.) This value corresponds to the lower calibration limit of the analytical method. Steps to correct the contamination must be taken whenever these levels are exceeded. To that effect, first vacuum the working places (hoods, benches, sink) using a vacuum cleaner equipped with a high-efficiency particulate absorbent (HEPA) filter and then wash with a detergent. A new set of wipes should be analyzed before anyone is allowed to work in the dioxin area of the laboratory.

The test results and the decontamination procedure must be reviewed with EH&S.

`Title: Preparation of Samples for Analysis of Polychlorinated Dioxins and Furans for Analysis HRGC/HRMS

[Methods 8290, 8290A & TO-9A]

Approvals (Signature/Date):

Elizabeth Nguyen Technical Manager — Date 12/21/12	Joe Schairer Health & Safety Manager / Coordinator — Date 12/20/12
Douglas Weir Quality Assurance Manager — Date 12/20/12	Karla Buechler Laboratory Director — Date 12/21/12

1. SCOPE AND APPLICATION

1.1. This method provides procedures for the preparation of samples prior to the analysis of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD), polychlorinated dibenzo-p-dioxins (tetra- through octachlorinated homologs; PCDDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologs; PCDFs) in a variety of environmental matrices at part-per-trillion (ppt) concentrations by SW 846 Method 8290. The analytical method calls for the use of high-resolution gas chromatography and high-resolution mass spectrometry (HRGC/HRMS) on purified sample extracts. Refer to Table 1 for the list of analytes. Analysis is by SOP WS-ID-0005.

1.2. The sensitivity of this method is dependent upon the level of interferences within a given matrix.

1.3. This method is designed for use by analysts who are experienced with residue analysis.

1.4. Samples containing concentrations of specific congeners (PCDDs and PCDFs) that are greater than the calibration limit should be analyzed by a protocol designed for such concentrations, such as 8280A/B.

2. SUMMARY OF METHOD

2.1. This procedure uses matrix-specific extraction and analyte-specific cleanup techniques.

2.2. A specified amount (see Table 1) of soil, sediment, fly ash, water, sludge (including paper pulp), still-bottom, fuel oil, chemical reactor residue, air sample (QFF, PUF or XAD media) or fish tissue, is spiked with a solution containing specified amounts of each of nine isotopically (^{13}C) labeled PCDDs/PCDFs listed in Table 2. The sample is then extracted according to a matrix-specified extraction procedure. The extraction procedures are: a) toluene Soxhlet (or equivalent) extraction, for soil, sediment, fly ash samples, aqueous sludges, and solid air matrices (XAD, QFF, PUF); b) methylene chloride liquid-liquid extraction or solid phase extraction for water samples; c) dilution of a small sample aliquot in solvent for wastes/chemical products; and d) toluene (or hexane/methylene chloride) Soxhlet (or equivalent) extraction for fish tissue. This method can also use solid phase extraction (SPE), however, Test America West Sacramento is in the developmental stages for this extraction type and is not currently certified for its use.

2.3. If interferences are present, extracts may be cleaned as described below. The extracts are submitted to an acid and/or base washing treatment and dried. Following a solvent exchange step, the residue is cleaned up by column chromatography on acid/base silica, acid alumina and carbon on silica. The preparation of the final extract for HRGC/HRMS analysis is accomplished by adding 20 µL of a tetradecane solution containing 100 pg/µL of each of the two recovery standards $^{13}C_{12}$-1,2,3,4-TCDD and

$^{13}C_{12}$ -1,2,3,7,8,9-HxCDD (Table 2) to the concentrated eluate. The former is used to determine the percent recoveries of tetra- and penta-chlorinated PCDD/PCDF internal standards while the latter is used for the determination of hexa-, hepta- and octa-chlorinated PCDD/PCDF internal standard percent recoveries. Upon client approval, less final volume can be used to decrease detection limit and more final volume can be used to decrease severe interferences.

3. DEFINITIONS

3.1. Definitions of terms used in this SOP may be found in the glossary of the Quality Assurance Manual (QAM).

3.2. Data qualifiers are defined on each data report. Commonly used data qualifiers are defined in the QAM.

3.3. Internal Standard: An internal standard is a ^{13}C-labeled analog of a congener chosen from the compounds listed in Table 2. Internal standards are added to all samples including method blanks and quality control samples before extraction, and they are used to quantitate the concentration of the analytes. Nine internal standards are used in this method. There is one for each of the dioxin and furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight. Additional internal standards may be added to act as retention time references, but they are not used for quantitation.

3.4. Recovery Standard: Two recovery standards are used to determine the percent recoveries for the internal standards. The $^{13}C_{12}$-1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated internal standards while $^{13}C_{12}$-1,2,3,7,8,9-HxCDD is used to determine the recovery of the hexa-hepta- and octachlorinated internal standards. $^{13}C_{12}$-1,2,3,7,8,9-HxCDD also acts as a retention time reference for the unlabeled analog present in sample extracts. They are added to the final sample extract before HRGC/HRMS instrument analysis.

3.5. Cleanup Recovery Standard (CRS): A $^{37}Cl_4$-2,3,7,8-TCDD analog that is added to each sample following extraction to measure the efficiency of the cleanup process.

4. INTERFERENCES

4.1. Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts or elevated baselines that may cause misinterpretation of the chromatographic data. All of these materials must be demonstrated to be free from interferents under the conditions of analysis by running laboratory method blanks. Analysts shall not use PVC gloves.

4.2. The use of high-purity reagents and solvents helps minimize interference problems. Purification of solvents by distillation in all-glass systems may be necessary.

4.3. Proper cleaning of glassware is extremely important because glassware may not only contaminate the samples, but may also remove the analytes of interest by adsorption on the glassware surface.

4.3.1. Glassware should be rinsed with solvent and washed with a detergent solution as soon after use as is practical. Sonication of glassware containing a detergent solution for approximately 30 seconds may aid in cleaning. Glassware with removable parts, particularly separatory funnels with Teflon stopcocks, must be disassembled prior to detergent washing.

4.3.2. After detergent washing, glassware should be immediately rinsed with acetone, toluene, hexane, and then methylene chloride.

4.3.3. Do not kiln reusable glassware in an oven as a routine part of cleaning. Kilning may be warranted after particularly dirty samples are encountered, but should be minimized, as repeated kilning of glassware may cause the formation of active sites on the glass surface that will irreversibly adsorb PCDDs/ PCDFs.

4.3.4. Immediately prior to use, Soxhlet (or equivalent) extraction glassware should be pre-extracted with toluene for a minimum of 3 hours. Note: Accelerated extractors such as the Soxtherm can use a shorter cleaning cycle which exhibits subsequent extractions free of cross contamination and interferences.

Note: Re-use of glassware should be minimized to avoid the risk of contamination. All glassware that is re-used must be scrupulously cleaned as soon as possible after use, applying the following procedure:

4.4. Interferences co-extracted from samples will vary considerably from source to source, depending on the diversity of the site being sampled. Interfering compounds may be present at concentrations several orders of magnitude higher than the PCDDs and PCDFs. The most frequently encountered interferences are chlorinated-biphenyls, methoxy biphenyls, hydroxy biphenyl ethers, benzyl phenyl ethers, polynuclear aromatics, and pesticides. Because very low levels of PCDDs and PCDFs are measured by this method, the elimination of interferences is essential. The cleanup steps given in Sections 11.12 thru 11.16 can be used to reduce or eliminate these interferences.

4.4.1. If South Carolina samples show diphenyl ethers at levels that could contribute to positive furan hits, a subsequent clean-up to remove them must be performed.

5. SAFETY

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), the West Sacramento Addendum to the Corporate EH&S Manual (WS-PEHS-002) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toes, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

5.1.1. Hearing protection must be worn when using mechanical systems to grind fish, tissue, or paper/pulp samples.

5.1.2. Finely divided dry soils contaminated with PCDDs and PCDFs are particularly hazardous because of the potential for inhalation and ingestion. Such samples are to be processed in a confined environment, such as a hood or a glove box.

5.1.3. Assembly and disassembly of glassware creates a risk of breakage and cuts. All staff members shall wear Kevlar or MAPA blue latex cut-resistant gloves over chemically resistant gloves when assembling and disassembling glassware.

5.1.4. Eye protection that satisfies ANSI Z87.1, laboratory coat, and chemically resistant gloves must be worn while samples, standards, solvents, and reagents are being handled. Latex and vinyl gloves provide no protection against most of the organic solvents used in this method. Nitrile or similar gloves must be used. Latex gloves may be used for methanol.

5.1.5. Exposure to chemicals must be maintained as low as reasonably achievable, therefore all samples must be opened, transferred and prepared in a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.1.6. Laboratory procedures such as repetitive use of pipets, repetitive transferring of extracts, and manipulation of filled separatory funnels and other glassware represent a significant potential for repetitive motion or other ergonomic injuries. Laboratory associates performing these procedures are in the best position to realize when they are at risk for these types of injuries. Whenever a situation is found in which an employee is performing the same repetitive motion, the employee shall immediately bring this to the attention of their supervisor, manager, or the EH&S staff. The task will be analyzed to determine a better means of accomplishing it.

5.1.7. The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. The use of separatory funnels during the partition and back extraction of sample extracts can also create excessive pressure. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggles when it is performed. Alternately, the extraction can be performed behind a closed fume hood sash on a mechanical shaker.

5.1.8. When Dean-Stark/Soxhlet clean-ups or extractions are performed overnight or unattended, special precautions must be taken. Open the chiller valves to the system about 15 minutes before the heating elements are turned on, and check every condenser to ensure that it is cold and functioning properly before turning the heating elements on. Check every condenser again about 15 minutes after turning the heating elements on to ensure that they are still cold and functioning properly. If the system is left operating overnight or unattended for an extended period, the first chemist to come back into the lab must again check every condenser to ensure that it is still cold and functioning properly.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Benzene	Flammable Toxic Carcinogen	PEL: 1 ppm TWA ; 5 ppm 15 MIN. STEL	Causes skin irritation. Toxic if absorbed through skin. Causes severe eye irritation. Toxic if inhaled. Vapor or mist causes irritation to mucous membranes and upper respiratory tract. Exposure can cause narcotic effect. Inhalation at high concentrations may have an initial stimulatory effect on the central nervous system characterized by exhilaration, nervous excitation and/or giddiness, depression, drowsiness or fatigue. Victim may experience tightness in the chest, breathlessness, and loss of consciousness.
Cyclohexane	Flammable Irritant	300 ppm TWA	Inhalation of vapors causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. High concentrations have a narcotic effect.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Isooctane	Flammable Irritant	None established	Inhalation of vapors may cause nausea, headache, dizziness, loss of consciousness, irritation to upper respiratory tract, pain in throat and nose, coughing, wheezing, shortness of breath.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid (1)	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Tetradecane	Irritant	None established	Inhalation of vapors may cause difficulty breathing, headache, intoxication and central nervous system damage.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

The following list of items does not necessarily constitute an exhaustive compendium of the equipment needed for this analytical method.

6.1. Nitrogen evaporation apparatus with variable flow rate.

6.2. Balances capable of accurately weighing to 0.01 g and 0.0001 g.

6.3. Centrifuge.

6.4. Water bath, equipped with concentric ring covers and capable of maintaining temperature control within ± 2ºC.

6.5. Stainless steel or glass containers large enough to hold contents of one-pint sample containers.

6.6. Drying oven.

6.7. Stainless steel spoons and spatulas.

6.8. Pipettes, disposable, Pasteur, 150 mm long x 5 mm ID.

6.9. Pipettes, disposable, serological, 10 mL, for the preparation of the carbon column specified in Section 7.1.

6.10. Reacti-vial, 2 mL, silanized clear glass.

6.11. Stainless steel meat grinder with a 3- to 5-mm hole size inner plate.

6.12. Separatory funnels, 250 mL.

6.13. Separatory funnels, 1000 mL.

6.14. Teflon® boiling chips (or equivalent) washed with methylene chloride before use.

6.15. Chromatographic column, glass, 300 mm x 10.5 mm, fitted with Teflon® stopcock.

6.16. Adapters for concentrator tubes.

6.17. Glass fiber filters, Whatman GF-D, GF-F, GMF150, or equivalent.

6.18. Solid phase extraction discs, 3M 90mm C18, or equivalent.

6.19. Dean-Stark trap, 5 or 10 mL, with T-joints, condenser and 125 mL flask.

6.20. Continuous liquid-liquid extractor.

6.21. All-glass Soxhlet apparatus, 500 mL flask.

6.22. Soxtherm extraction apparatus (or equivalent), including glass thimble holders, glass beakers, and gaskets.

6.23. Glass funnels, sized to hold 170 mL of liquid.

6.24. Desiccator.

6.25. Turbo evaporator

6.26. Rotary evaporator with a temperature controlled water bath.

6.27. High speed tissue homogenizer, equipped with an EN-8 probe or equivalent.

6.28. Glass wool, extracted with methylene chloride, dried and stored in a clean glass jar.

6.29. Vacuum extraction device for solid phase extraction, 1 Liter glass funnel with 90mm filter disc holder with a vacuum source, Kontes or equivalent.

7. REAGENTS AND STANDARDS

7.1. Column Chromatography Reagents

7.1.1. Silica Gel - Kieselgel 60 or equivalent, activate for 1 hour at 184°C before use. Store at 130°C in covered flask.

7.1.2. Acid Alumina - ICN or equivalent, activated as necessary.

7.1.3. Basic Alumina - ICN or equivalent. No activation required.

7.1.4. Granular carbon/silica gel - Mix 3.6 g granular carbon and 16.4 g activated silica gel; (alternatively, prepare carbon/silica gel (5%/95%); i.e., combine 5 g precleaned carbon with 95 g silica gel). Store at room temperature in a Teflon ® lined covered jar. The first LCS prepared with a new batch of column packing material is the quality control check of the packing materials. Refer to historical control limits before accepting the new batch of material.

7.1.5. 44% H_2SO_4 /silica gel - Mix 24 mL conc. H_2SO_4 and 56 g activated silica gel. Stir and shake until free flowing. Store at room temperature.

7.1.6. 33% NaOH/silica gel - Mix 34 mL 1N NaOH and 67 g activated silica gel. Stir and shake until free flowing. Store at room temperature.

7.2. Acid Alumina Activity Assessment

Alumina activity may vary with the matrix or environmental conditions. Monitor internal standard and cleanup recovery standard recoveries in extract analysis. Low recoveries of cleanup recovery standard (CRS) may indicate loss of alumina activity. Assess stability of alumina activity and apply corrective action as appropriate (reactivate and reprofile).

Note: a column profile should be done to show elution of all 2,3,7,8 substituted analogs so problems can be readily identified.

7.2.1. Profile each vendor lot of activated alumina as corrective action for low internal standard and CRS recoveries dictate. If necessary, proceed as follows:

7.2.1.1. Set up and label 3 acid alumina columns.

7.2.1.2. Pre-rinse with 20 mL hexane.

7.2.1.3. Add 2 mL hexane spiked with internal standards and natives (spike amounts equivalent to those for LCS) with 2X2 mL hexane rinse of fractions.

7.2.1.4. Elute each column with 20 mL hexane. Collect and label these fractions.

7.2.1.5. Elute each column with 5 x10 mL methylene chloride/hexane at the appropriate v/v percent. Collect and label these fractions separately.

7.2.1.6. Elute each column with 10 mL of 100% methylene chloride. Collect and label these fractions. Reduce all fractions to final volume and add recovery standard.

7.2.2. Review data and select an elution scheme. Group the fraction from each solvent system as follows:

7.2.2.1. Pre-analyte fraction - consists of all eluent prior to elution of first target analytes.

7.2.2.2. Analyte fraction - consists of all that contain detectable levels of target analytes.

7.2.2.3. Post-analyte fraction - consists of all eluents after elution of the last target analyte.

7.2.3. Select the solvent system which best meets the following two conditions:

7.2.3.1. Pre-analyte fraction consists of 20mL hexane and no more than 20 mL mixed solvent.

7.2.3.2. Analyte fraction consists of no more than 20mL of mixed solvent and contains greater than 90% of all target analytes and greater than 80% of all internal standards.

7.2.4. After selection of the appropriate solvent system and fractionation pattern, perform triplicate acid alumina cleanups on spiked hexane to ensure reproducibility of the fractionation pattern. Document each elution scheme.

7.2.5. Each subsequent batch of acid alumina used in the lab (from the same vendor lot) must be checked for stable activity.

7.3. Reagents

7.3.1. Sulfuric acid, concentrated, ACS grade, specific gravity 1.84.

7.3.2. Distilled water demonstrated to be free of interferents

7.3.3. 1 N HCl.

7.3.4. Silica gel.

7.3.5. Solution for breaking emulsions: Slowly add 1.0L of reagent grade NaOH solution to a 2.0L NaOH container, containing 1.0L of DI H2O, and leave the container in secondary containment with the lid off.

Warning: The solution will begin to heat so let the solution stand until equilibrium is met and the solution is at room temperature.

When this process is complete, the solution will then be ready for use in the samples.

7.3.6. Precleaned Sodium Sulfate.

7.3.7. Canola Oil (for tissue extraction only), or other suitable oil.

7.4. Desiccating Agent

7.4.1. Sodium sulfate, granular, anhydrous.

7.5. Solvents

7.5.1. High-purity, distilled-in-glass or highest available purity: Methylene chloride, hexane, methanol, tetradecane, isooctane, toluene, cyclohexane, and acetone.

7.6. All daily internal standard, daily clean up recovery standards, and daily spiking solutions are stable for one year from preparation. After 1 year, solutions may be re-verified. The re-verified solution may be used for an additional year, or until there is evidence of compound degradation or concentration. The re-verification must be performed using an unexpired, not previously re-verified solution from a second lot or second vendor.

7.6.1. Sealed ampules may be used until the manufacturer's expiration date is exceeded. If no expiration date is provided, then the expiration date will be 10 years from the date the ampule is opened. The solvent level should be monitored prior to each use to assure there has been no concentration of the standard over time.

7.6.2. Standards for method 8290A require storage at ≤ 6ºC.

7.7. Field Surrogate Solution (air matrices)

This solution contains one ^{37}Cl labeled analog (for Method TO-9/TO-9A) or one ^{37}Cl and four ^{13}C labeled analogs (for Method 0023) at the nominal concentration indicated in Table 2. It is used to assess sample collection and recovery procedures.

7.8. Internal Standard

This isooctane solution contains the nine internal standards at the nominal concentrations that are listed in Table 2. The solution contains at least one carbon-labeled standard for each homologous series, and it is used to measure the concentrations of the native substances. (Note that $^{13}C_{12}$ -OCDF is not present in the solution.)

7.9. Native Spike Standard

Also known as the Matrix Spike or Native Spike solution. Contains all the 2,3,7,8-substituted unlabeled analytes listed in Table 2. Prepare using the appropriate standards to yield a spiking solution with a concentration of 4.0 ng/ml for the tetra-CDDs/CDFs, 20 ng/ml for the penta-, hexa-, and hepta- CDDs/CDFs, and 40 ng/ml for the octa- CDD/CDF.

7.10. Recovery Standard Solution

This tetradecane solution contains two recovery standards ($^{13}C_{12}$-1,2,3,4-TCDD and $^{13}C_{12}$ -1,2,3,7,8,HxCDD). An appropriate volume of this solution is spiked into each sample extract before the final concentration step.

7.11. Cleanup Recovery Standard Solution (CRS)

Prepare $^{37}Cl_4$-2,3,7,8-TCDD at the concentration shown in Table 2, in isooctane (or toluene).

7.12. Preparation and QC of PUF material

7.12.1. The PUF material is purchased pre-cut.

7.12.2. The PUFs are rinsed by Soxhlet with acetone (or other appropriate solvent) for a minimum of 16 hours and air dried for a minimum of 2 hours in a contaminant-free area.

7.12.3. One PUF from the rinsed batch is randomly selected to be the QC sample for the batch.

7.12.4. The PUF is loaded into a pre-cleaned Soxhlet extractor charged with toluene.

7.12.5. The 1613/8290 daily internal standard solution is spiked into the PUF and it

is extracted for a minimum of 16 hours.

7.12.6. The Soxhlet extract is recovered and processed according to Section 11.4.

7.12.7. The batch of PUF is considered acceptable if no target analytes are detected at or above the laboratory or project specific reporting limit.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. The sample collection, shipping, handling, and chain-of-custody procedures are not described in this document. Sample collection personnel will, to the extent possible, homogenize samples in the field before filling the sample containers. This should minimize or eliminate the necessity for sample homogenization in the laboratory. The analyst should make a judgment, based on the appearance of the sample, regarding the necessity for additional mixing. If the sample is clearly non-homogeneous, the entire contents should be transferred to a glass or stainless steel pan for mixing with a stainless steel spoon or spatula before removal of a sample portion for analysis.

8.2. Grab and composite samples must be collected in glass containers.

8.3. Ambient air samples are collected on a Quartz Fiber Filter followed by a glass sleeve containing a polyurethane foam plug.

8.4. Samples from stationary sources are collected on glass or quartz fiber filters and XAD-2 Resin. (See WS-ID-0009 for sample preparation procedures).

8.5. Conventional sampling practices must be followed. Do not rinse the bottle with sample before collection. Sampling equipment must be free of potential sources of contamination.

8.6. Grinding or blending of fish samples.

If not otherwise specified by the client, the whole fish (frozen) should be blended or ground to provide a homogeneous sample. The use of a stainless steel meat grinder with a 3 to 5 mm hole size inner plate is recommended. In some circumstances, analysis of fillet or specific organs of fish may be requested by the client. If so requested by the client, the above whole fish requirement is superseded. More detail can be found in "Tissue Sampling and Handling for a variety of Methods" (WS-WI-0018).

Warning: Hearing protection must be worn when grinding samples.

8.7. With the exception of the fish tissues, which must be stored at - 20°C, all samples should be stored at 4°C ± 2, extracted within 30 days and completely analyzed within 45 days of collection. The 30 day hold time is recommended. PCDDs and PCDFs have demonstrated stability for greater than one year.

8.8. All extracts must be stored capped, in the dark, at room temperature (approximately 21°C to 28°C). All extracts for method 8290A must be stored capped at ≤ 6ºC.

8.9. For moisture determinations refer to SOP WS-OP-0013.

9. QUALITY CONTROL

9.1. One method blank (MB) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The method blank is an aliquot of laboratory matrix (reagent water, sodium sulfate, PUF, XAD, filter, etc.) processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when target analytes are detected in the method blank above the reporting limit or when surrogate recoveries are outside control limits. Re-extraction of the blank, other batch QC, and the affected samples are required when the method blank is deemed unacceptable. The method blank contains a PUF plug, XAD, or filter prepared from the same batch as the field samples whenever possible for air samples.

Certain programs, such as DOD, may require a more stringent evaluation of the method blank, for instance, that the blank not contain any analytes of interest at a concentration greater than ½ the lower calibration limit.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.1.1. If the accompanying samples are aqueous, use distilled water as a matrix. Take the method blank through all steps detailed in the analytical procedure.

9.1.2. Use sodium sulfate as the method laboratory matrix when solids are extracted. Use a mixture of sodium sulfate and canola oil as the matrix when tissues are extracted. Take the method blank through all steps detailed in the analytical procedure.

9.1.3. The method blank must be spiked prior to extraction with the same amount of ^{13}C -labeled internal standards as added to samples.

9.1.4. If method blank contamination is present, check solvents, reagents, fortification solutions, apparatus and glassware to locate and eliminate the source of contamination before any further samples are extracted and analyzed. The presence of any analyte in the method blank ate concentrations greater than the reporting limit (RL) is cause for corrective action.

9.1.4.1. OCDD is a ubiquitous laboratory contaminant. A method blank and the associated samples are deemed acceptable if the OCDD

concentration is <5x the specified reporting limit. Flag data appropriately. The analyst is expected to investigate and eliminate potential sources of systematic contamination.

9.1.4.2. If a target analyte is detected in the blank but the associated samples are ND (not detected), then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.4.3. If a target analyte is detected in the blank, but the concentration of the contaminant in the samples >10x the blank concentration, then the data may be reported, unless otherwise directed by the client. Note the action in the narrative.

9.1.4.4. If one of the conditions above is not met then the sample associated with a contaminated method blank must be re-extracted.

9.1.5. If new batches of reagents or solvents contain interfering contaminants, purify or discard them.

9.2. A Laboratory Control Sample (LCS) must be extracted with every process batch of similar matrix, not to exceed twenty (20) samples. The LCS is an aliquot of laboratory matrix (e.g. water, sodium sulfate, PUF, XAD, etc.) spiked with analytes of known identity and concentration. The LCS must be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spiked analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, other batch QC and all associated samples are required if the LCS is deemed unacceptable. See policy WS-PQA-003 for specific acceptance criteria. When associated with PUF samples, the LCS should contain a PUF plug prepared from the same batch as the field samples whenever possible.

Note: Re-extraction of the blank, QC and affected samples for the air matrices (PUF, XAD, and filter) is not generally possible because the entire sample is consumed in the initial extraction.

9.2.1. A LCS is deemed acceptable if control analytes are above control limits and the associated samples are ND, unless otherwise specified by the client. Note any actions in the narrative.

9.3. The assessment of matrix effects on method performance, as required by NELAP, is met in Method 8290 and 8290A, as in all isotope dilution techniques, with the use of isotopically labeled compounds. These isotopically labeled compounds are analogs of target analytes and are spiked into each sample. Therefore, matrix effects on method performance may be judged by the recovery of these analogs. Sample analysis

acceptance is controlled by the performance of these analogs in each sample. A Matrix Spike/Matrix Spike Duplicate (MS/MSD or MS/SD) pair are extracted at the client's request only. Method 8290A does not address analysis of MS/MSD. An exception to this rule is a batch containing South Carolina samples for Method 8290. These batches must have an MS/MSD prepared. However, South Carolina requires Method 8290A after December 31, 2008. An MS/MSD pair are aliquots of a selected field sample spiked with analytes of known identity and concentration. When requested by the client, the MS/MSD pair shall be processed in the same manner and at the same time as the associated samples. Corrective actions must be documented on a Non-Conformance memo, then implemented when recoveries of any spike analyte is outside control limits provided on the LIMS or by the client. Re-extraction of the blank, the LCS, the selected field sample, and the MS/MSD may be required after evaluation and review. Matrix Spike/ Matrix Spike Duplicates are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. An LCS/LCSD may be extracted to show precision of the extraction and analysis process.

9.3.1. Matrix Spike (MS): A sample, which is spiked with a known amount of the matrix spike fortification solution prior to the extraction step. The recoveries of the matrix spike compounds are determined; they are used to estimate the effect of the sample matrix upon the analytical methodology.

9.3.2. Matrix Spike Duplicate (MSD): A second portion of the same sample as used in the matrix spike analysis and which is treated like the matrix spike sample.

9.3.3. Locate the sample for the MS and MSD analyses (the sample may be labeled "double volume").

9.3.4. Add an appropriate volume of the matrix spike fortification solution, adjusting the fortification level as specified in Table 1, under IS Spiking Levels.

9.3.5. The results obtained from the MS and MSD samples (percent recovery and concentrations of 2,3,7,8-substituted PCDDs/PCDFs) should agree within 20 percent relative difference. Report all results and flag outliers.

9.3.6. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.4. Duplicates

9.4.1. Upon client request, duplicates may be processed. Locate the sample specified for duplicate analysis, and prepare and analyze a second 10-g soil or sediment sample portion or 1 L water sample, or an appropriate amount of

the type of matrix under consideration. Duplicate samples are not generally applicable for air samples due to the difficulty in collecting identical or representative samples. A duplicate injection of a sample extract may be performed to display instrument precision.

9.4.1.1. The results of the laboratory duplicates (percent recovery and concentrations of 2,3,7,8-substituted PCDD/PCDF compounds) should agree within 25 percent relative difference. Report all results and flag outliers.

9.4.2. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.5. Field Blanks

9.5.1. Each batch of samples may contain a field blank sample of nominally uncontaminated soil, sediment or water that is to be processed for analysis.

9.5.1.1. Weigh a 10-g portion or use 1 L (for aqueous samples) of the specified field blank sample and add the appropriate amount of internal standard to yield 100 pg/µL in the final extract.

9.5.1.2. Extract by using the procedures described in Section 11. As applicable, add the appropriate amount of recovery standard to yield 100 pg/µL in the final extract. Analyze a 1-2 µL aliquot of the concentrated extract using SOP WS-ID-0005.

9.6. Rinsate Samples

9.6.1. In addition to the field blank, a batch of samples may include a rinsate, which is a portion of the solvent (usually trichloroethylene) that was used to rinse sampling equipment. The rinsate is analyzed to assure that the samples were not contaminated by the sampling equipment.

9.6.2. The rinsate sample must be processed like a regular sample.

Take a 100-mL (± 0.5 mL) portion of the sampling equipment rinse solvent (rinsate sample), filter, if necessary, and add the appropriate amount of internal standard to yield 100 pg/µL in the final extract.

9.6.3. Using appropriate methods, concentrate to approximately 10 mL.

9.6.4. Just before analysis, add the appropriate amount of recovery standard to yield 100 pg/µL in the final extract. Reduce the volume to a final volume of 20 µL, as necessary. No column chromatography is required.

9.6.5. Analyze an aliquot following the same procedures used to analyze samples.

9.7. Surrogate/Clean Up Recovery Standard

A surrogate compound may be spiked into all air media samples prior to collection. For all other matrices, a clean up recovery standard is spiked following extraction and just prior to cleanup, in order to monitor relative loss of internal standard during both extraction and cleanup.

9.8. Internal Standards

An internal standard is a ^{13}C -labeled analog of a PCDD/PCDF congener. Internal standards are added to all samples including method blanks and quality control samples before extraction, and they are used to quantitate the concentration of the analytes. Nine internal standards are used in this method. There is one for each of the dioxin and furan homologs (except for OCDF) with the degree of chlorination ranging from four to eight. Additional internal standards may be added to act as retention time references, but they are not used for quantitation.

9.8.1. A 2000 pg aliquot of the internal standard mixture is added to all samples, regardless of sample size. As an example, for $^{13}C_{12}$ -2,3,7,8-TCDD, a 10-g soil sample requires the addition of 2000 pg of $^{13}C_{12}$ -2,3,7,8-TCDD to give the requisite fortification level.

9.8.2. Internal standards must be spiked into all samples, QC samples, and included in all calibrations.

9.8.3. For each sample and QC aliquot, calculate the percent recovery. The percent recovery should be between 40 percent and 135 percent for all nine internal standards.

9.8.4. A low or high percent recovery for a blank does not require discarding the analytical data but it may indicate a potential problem with future analytical data. Internal standard recoveries are flagged if they are outside the recovery goals. Re-extraction of affected samples should be performed if signal-to-noise for any internal standard is less than 10:1.

9.9. Recovery Standard: Two recovery standards are used to determine the percent recoveries for the internal standards. The $^{13}C_{12}$ -1,2,3,4-TCDD is used to measure the percent recoveries of the tetra- and pentachlorinated internal standards while $^{13}C_{12}$ -1,2,3,7,8,9-HxCDD is used to determine the recovery of the hexa-hepta- and octachlorinated internal standards. $^{13}C_{12}$ -1,2,3,7,8,9-HxCDD also acts as a retention time reference for the unlabeled analog present in sample extracts. They are added to the final sample extract before HRGC/HRMS instrument analysis.

9.10. Recommended Corrective Actions and Troubleshooting Steps

- Verify satisfactory instrument performance.
- If possible, verify that no error was made while weighing the sample aliquots.
- Review the analytical procedures with the performing laboratory personnel.

10. CALIBRATION

10.1. On a daily basis, calibrate any balance to be used in accordance with SOP WS-QA-0041.

10.2. On a monthly basis, calibrate any autopipettor to be used in accordance with SOP WS-QA-0004.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of the supervisor to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2. Refer to SOP WS-ID-0009 for the preparation of stationary source samples.

11.3. Sample Pre-Treatment

11.3.1. Paper Pulp Sludges are generally air-dried and ground prior to extraction following Section 11.5. Because of the drying procedure, a Dean-Stark water separator is optional for extraction.

11.3.2. Fly Ash — Fly ash samples are pretreated with HCl prior to extraction by both soxhlet and separatory funnel techniques.

11.3.2.1. Weigh 2-10g of sample aliquot into a clean glass jar.

11.3.2.2. Add 1.0mL of the internal standard mixture with 2 mL of acetone.

11.3.2.3. Add 150 mL of 1N hydrochloric acid and shake for 4 hours.

11.3.2.4. If the sample reacts violently with acid, then allow the sample to equilibrate for 4 hours with no shaking.

11.3.2.5. Filter the contents of the jar through a glass fiber filter.

11.3.2.6. Extract the solids as per Section 11.5, omitting the daily internal standard spike for the samples.

11.3.2.7. Extract the aqueous filtrate as per Section 11.8, using 100 mL of toluene for the first shake, and 100 mL of hexane for subsequent shakes.

11.3.2.8. Concentrate the combined toluene solutions to near dryness on a rotary evaporator at 50°C. Proceed with Section 11.12 as necessary.

Note: As an option, a Soxhlet/Dean Stark extractor system may be used, with toluene as the solvent. No sodium sulfate is added when using this option.

11.4. Waste Dilution (Still-Bottom/Fuel Oil, and other solvent-miscible materials).

11.4.1. Weigh 1 g of the waste (organic liquids, fuel oils, and solids that will dissolve in a solvent) into a vial.

11.4.2. Add 40 mL of toluene (or other solvent if the material is not miscible/soluble in toluene). Shake gently to dissolve.

11.4.3. Remove a 4.0 mL aliquot (0.1g sample equivalent) and place in a culture tube. Add 1.0 mL of daily internal standard and 1.0 mL of cleanup recovery standard, and proceed to Section 11.12.

11.5. Soxhlet Extraction (Solids, Tissues, Sludges, Wipes)

11.5.1. Pre-extract the glassware by heating the flask until the toluene is boiling. When properly adjusted, 1-2 drops of toluene per second will fall from the condenser tip into the receiver. Extract the apparatus for a minimum of four hours.

WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensors are cold before you turn the heating element on. Check all of the condensors about 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If this cleaning cycle is to be left unattended (e.g., overnight) the first chemist to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.

11.5.2. After pre-extraction, cool and disassemble the apparatus.

11.5.3. If tissues requiring % Lipids are to be extracted, for each sample weigh the concentration vessel with label and boiling chips. Record the mass on the benchsheet. Refer to SOP WS-QA-0018 “Subsampling”, for instructions on how to homogenize and subsample the container of sample.

11.5.4. Weigh a well-mixed aliquot of each sample (10 g, unless otherwise specified) into a clean Soxhlet thimble. Record the mass to the nearest 0.01g. Use sodium sulfate for the batch QC (MB, LCS) for solids, and a mixture of 9 g sodium sulfate and 1 g canola oil for the batch QC for tissue matrices.

11.5.4.1. In the case of wipes, place the entire wipe sample into the Soxhlet apparatus (no thimble needed), including any liquid present with the sample. Use pre-cleaned wipes for the batch QC samples.

11.5.5. Place the thimble into a Soxhlet apparatus equipped with a Dean-Stark water separator.

11.5.6. Spike all samples with 1.0 mL of internal standard solution (2 pg/µL), for a final concentration of 200 pg/g (based on a 10 g sample).

11.5.7. Spike the LCS (and MS/MSD, if present) with 50 uL of native spike.

11.5.8. Reassemble the pre-extracted apparatus and add a fresh charge (250-300 mL) of toluene to the receiver and reflux flask.

11.5.9. Reflux 16 hours, with the solvent cycling at least 5 times per hour.

WARNING: Open the chiller supply valves about 15 minutes before turning on the heating element and ensure that all of the condensors are cold before you turn the heating element on. Check all of the condensors about 15 minutes after starting the heating process to ensure that they are still cold and functioning properly. If this cleaning cycle is to be left unattended (e.g., overnight) the first chemist to arrive the next morning is to check all condensers to ensure that they are still cold and functioning properly.

11.5.10. Drain the water from the receiver if the receiver fills with water. Check and drain when necessary.

Note: If the receiver holds 10 mL of liquid, and 20 g of an approximately 10% solid sample is being extracted, then approximately 9 mL of water will end up in the receiver. In this case, the receiver will not need to be emptied (insufficient liquid to overflow), but it should be checked. If the sample amount is 50, and the percent solids is still 10%, then 45 mL of water will end up in the receiver. In this case, frequent checking is required, and the receiver will need to be emptied at least 5 times.

11.5.11. After refluxing, allow the apparatus to cool.

11.5.12. If samples DO NOT require % lipids add 100 µL of tetradecane as a keeper to the round bottom flask.

11.5.13. Proceed to Section 11.17.

11.6. SoxTherm Extraction (Solids, Tissues, Sludges,Wipes)

11.6.1. Prior to loading samples, run the system through 2 cleaning cycles (approximately 1 hour each).

11.6.2. After pre-extraction, cool and disassemble the apparatus.

11.6.3. Weigh a well-mixed aliquot of each sample (10 g, unless otherwise specified) into a clean Soxhlet thimble. Record the mass to the nearest 0.01g. Use sodium sulfate for the batch QC (MB, LCS) for solids, and a mixture of 9 g sodium sulfate and 1 g canola oil for the batch QC for tissue matrices.

11.6.3.1. In the case of wipes, place the entire wipe sample into the Soxhlet apparatus (no thimble needed), including any liquid present with the sample. Use pre-cleaned wipes for the batch QC samples.

11.6.4. Place the thimble into the Soxtherm apparatus.

11.6.5. Spike all samples with 1.0 mL of internal standard solution (2 pg/µL), for a final concentration of 200 pg/g (based on a 10 g sample).

11.6.6. Spike the LCS (and MS/MSD, if present) with 50 uL of native spike.

11.6.7. Reassemble the pre-extracted apparatus and add a fresh charge (150 mL) of toluene to the apparatus.

11.6.8. Program the system to boil for 1 hour, and reduce the toluene volume by 70-90 mL (volume < volume of the thimble).

11.6.9. Continue the extraction for one hour fifteen minutes, reducing the toluene volume by another 15 mL.

11.6.10. After refluxing, allow the apparatus to cool.

11.6.11. Pour the samples into round bottom flasks, and if samples DO NOT require % lipids add 100 µL of tetradecane as a keeper to the round bottom flask.

11.6.12. Proceed to Section 11.17.

11.7. Extract Splitting (Wipes)

Wipe extracts prepared using either Soxhlet or shaking techniques are split prior to further workup, to permit an archive aliquot, or analysis by an additional method.

Once the extract has been concentrated using the rotovap or Turbovap, proceed as follows:

11.7.1. Add approximately 1 mL of hexane or toluene to rinse the sides of the round bottom flask. Using a pipette, withdraw the sample from the round bottom flask and transfer the liquid into a test-tube. Use additional amounts of solvents to rinse the flask. Transfer all the liquid into the test-tube. Ensure that all traces of sample in the round bottom flask have been thoroughly rinsed from all surfaces. Bring the sample volume to 8.0 mL or 10.0 mL (or appropriate volume) with the addition of rinse solvent.

11.7.2. Upon completion of the rinsing, cap the test tube and shake vigorously. Take ½ of each sample (or an appropriate amount as instructed by the client, program manager or department manager) and transfer to a culture tube. Archive the remaining sample for future use.

11.7.2.1. If only one analysis is required, then ½ of the sample is archived and the other half is analyzed.

11.7.2.2. If "N" analyses are required, then the extract is divided into "N+1" equal portions, so that one portion is archived, and a portion is used for each test.

11.8. Aqueous Samples (liquid/liquid extraction).

11.8.1. When setting up the glassware for a batch, for each sample label one separatory funnel and one 500 mL round-bottom flask with the sample ID.

11.8.2. Weigh the sample in the bottle on the top loading balance to the nearest centigram (0.01g), and record the mass.

11.8.3. For each sample, add 1 mL of daily internal standard solution into 2 mL of acetone. Add this solution to the sample in the separatory funnel. Each aliquot of spike mixture is added similarly.

11.8.4. Dissolve 50µL of the target analyte into acetone and add this mixture into the LCS container.

11.8.5. Pour the entire sample (approximately 1L) into a 2L separatory funnel that is labeled with the sample ID.

11.8.6. Add 100 mL methylene chloride to the sample bottle, seal, and shake for 30 seconds to rinse the inner surface. Transfer the solvent to the separatory funnel.

11.8.7. Create a blank and LCS by adding 1 L of laboratory reagent water to 2

additional separatory funnels. Add 100 mL methylene choride to each funnel.

11.8.8. To the LCS, add 50 µL of the precision and recovery standard dissolved into 2 mL of acetone.

11.8.9. Extract the samples by shaking each funnel for two minutes with periodic venting.

Warning: Separatory funnel extraction with methylene chloride is a high-risk activity. Pressure may build rapidly in the funnel. It should be vented after several seconds of shaking, and often enough to prevent build-up of pressure. Chemist performing separatory funnel extraction must wear a face shield over their safety glasses/goggles. Alternatively, the extraction can be performed behind a closed fume hood sash.

11.8.10. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation.

11.8.11. Repeat the extraction two additional times with methylene chloride.

11.8.12. Determine the original sample volume by re-weighing the sample bottle. Record the sample volume to the nearest centigram (0.01g).

11.8.13. Dry extract with sodium sulfate: Place glass wool in a precleaned filter funnel. Rinse glass wool with methylene chloride and load funnel with Na_2SO_4. Pour extract through Na_2SO_4 to remove water. Rinse Na_2SO with fresh methylene chloride and collect in round bottom flask.

11.8.14. Transfer the extract to a 500 mL round-bottom previously labeled with the sample ID, then add approximately 100 µL of tetradecane and concentrate on a rotary evaporator or TurboVap.

11.8.15. Perform macro-concentration as detailed in Section 11.17.

11.9. Aqueous Samples (solid phase extraction).

11.9.1. Weigh the sample in the bottle on the top loading balance to the nearest centigram (0.01g), and record the mass.

11.9.2. Create a blank and LCS by adding 1L of laboratory reagent water to 2 additional 1L bottles.

11.9.3. For each sample, add 1mL of daily internal standard solution in acetone.

Add this solution to the sample in the bottles. Each aliquot of spike mixture is added similarly.

11.9.4. To the LCS, add 50µL of the precision and recovery standard in acetone.

11.9.5. Prepare the C18 extraction discs by first soaking them in toluene for at least 5 minutes.

11.9.6. Assemble the filter holder and vacuum filtration flask and place the extraction disc onto the filter holder. Place a GF-F filter on top of the extraction disc. If the sample has a large amount of particulates a GF-D filter can be placed on top of the GF-F filter. Alternatively, a GMF-150 filter can be used in place of the two filters.

11.9.7. Place the filtering funnel onto the disc holder and clamp it in place.

11.9.8. Rinse the filter and discs with approximately 15mL of toluene and allow it to soak for about a minute. Apply vacuum and draw the toluene through the discs. Repeat the wash step using about 15mL of acetone. Apply vacuum and draw the acetone through the discs.

11.9.9. Rinse the filter and discs with approximately 15mL of methanol and allow it to soak for about a minute. Apply vacuum and draw the methanol through the discs, but **DO NOT ALLOW THE DISCS TO GO DRY**. If they do go dry, simply repeat the methanol rinse step, leaving a 1 – 2mm layer of solvent on top of the discs.

11.9.10. Rinse twice with about 50mL of reagent water, leaving a 1 – 2mm layer of water on the surface of the discs.

11.9.11. Pour the spiked method blank, LCS or sample into the reservoir and apply vacuum to begin the extraction. Adjust the vacuum such that the extraction takes approximately 10 minutes. Samples with large amounts of particulates may take much longer.

11.9.12. After most of the sample has been pulled through the discs, rinse the sample bottle with a few mLs of reagent water and add the rinse to the funnel. Rinse down the sides of the funnel with reagent water as well.

11.9.13. Allow the discs to dry, remove them from the holder and extract by soxhlet (11.5) or soxtherm (11.6) and proceed with cleanups.

11.9.14. Determine the original sample volume by re-weighing the sample bottle. Record the sample volume to the nearest centigram (0.01g).

11.10. Breaking Emulsions

There are several useful methods to decrease or eliminate emulsion in aqueous samples when extracting with methylene chloride. These methods may include stirring with a pipette to manually breakup the emulsions or to transfer the sample into centrifuge tubes and centrifuge at approximately 3000 RPM. The most useful method is to use a 10:1 $NaOH/H_2O$ solution to change the pH enough to disrupt the emulsion phase, which works 90% of the time. See Section 7.3.5 for reagent preparation.

11.10.1. Check the pH of the sample to verify that the pH is between 3 and 7. If the pH is greater than 7, consult the supervisor and client for instructions.

11.10.2. Pour approximately 100 mL of the 10:1 $NaOH/H_2O$ into a 1 L amber glass bottle (AGB).

11.10.3. Drain the sample with the emulsion from the 2 L separatory funnel into the 1 L AGB and let it stand.

11.10.4. Empty the aqueous waste into the LLE waste drum.

11.10.5. Pour the solution with methylene chloride back into the same 2 L separatory funnel and drain the methylene chloride phase through Na_2SO_4 into a 500 mL round-bottom flask.

11.10.6. Empty the aqueous waste into the LLE waste drum.

11.10.7. Proceed with macro-concentration (Section 11.17).

11.11. Filter/PUF Samples

11.11.1. Place the glass sleeve containing the PUF and the Quartz Fiber Filter into the pre-cleaned Soxhlet extractor charged with toluene.

11.11.2. Add 2 mL (4000 pg) of 1613/8290 daily Internal Standard solution to all samples and QC.

11.11.3. Add 50 uL of 1613/8290 Native Spike to the LCS.

11.11.4. Extract the samples and QC for a minimum of 16 hours.

11.11.5. Concentrate the extract from the round bottom flask with hexane and adjust the volume.

11.11.6. Transfer the extract from the round bottom flask with hexane and adjust the volume.

11.11.7. Split the extract 50:50 for analysis and archive.

11.11.8. Proceed to Section 11.12.

11.12. Extract Clean-Up

11.12.1. For all samples that are not air media, spike 1.0 mL of the Cleanup Recovery Standard (CRS) prior to any cleanup into the round bottom flasks containing the samples and QC Extracts (See also Section 9.7).

11.12.2. Proceed with further cleanups as dictated by the sample matrix and extract color. The "Option C" cleanup (Section 11.13) and the IFB Upper Column cleanup (Section 11.14) are applied to samples with high levels of interferences. The IFB column cleanup (Section 11.15) is applied to all samples.

11.13. Acid Partitioning ("Option C")

11.13.1. Use this clean up as needed on samples with high levels of interferences. Consult with a lead chemist or department manager to determine applicability.

11.13.2. Partition the extract in 50-125 mL of hexane against 40 mL concentrated H_2SO_4 in a separatory funnel. Shake for two minutes. Remove and discard the H_2SO_4 layer (bottom). Repeat the acid washing until no color is visible in the acid layer (perform a maximum of four acid washings).

Warning: Shaking with a concentrated caustic is a high-risk activity. Analyst must wear a face shield over safety glasses/goggles, or the shaking must take behind a closed hood sash.

11.13.3. Partition the extract against 50 mL of distilled H_2O. Shake for two minutes. Remove and discard the aqueous layer (bottom). Dry the extract by pouring it through a funnel containing anhydrous sodium sulfate and collect it in a round-bottom flask. Rinse the sodium sulfate with two 15 mL portions of hexane, add the rinsates to the flask, and concentrate the hexane solution to near dryness on a rotary evaporator (35°C water bath), making sure all traces of toluene (when applicable) are removed. (Use of blow-down with an inert gas to concentrate the extract is also permitted.) The DI H_20 partition is applied only as samples warrant it at the discretion of the analyst.

11.14. IFB Upper Column Cleanup

11.14.1. Use this clean up as needed on samples with high levels of interferences. Consult with a lead chemist or department manager to determine applicability.

11.14.2. Set up the upper of the two chromatography columns as depicted in Figure 2.

The column (20 mm diameter) is packed in this order: a glass wool plug, 2 g activated silica gel, 4 g Acid silica gel, 2 g activated silica gel, and 1 g sodium sulfate.

11.14.3. Pre-rinse the column with 20 mL hexane, and discard the rinsate.

11.14.4. Add extract to the column. Rinse extract vessel 2 times with 1 mL each of hexane and add to column.

11.14.5. Elute 60 mL hexane directly onto acid silica column (upper column).

11.14.6. Collect the eluate, and concentrate before proceeding with the IFB cleanup (Section 11.15).

11.15. IFB Column Cleanup

Most samples will undergo this cleanup, either direction following concentration on the rotovap, or following the cleanup in Section 11.13 (Option C) or Section 11.14 (IFB Upper Column).

11.15.1. Set up two chromatography columns as depicted in Figure 2. The upper column (20 mm diameter) is packed in this order: a glass wool plug, 2 g activated silica gel, 4 g Acid silica gel, 2 g activated silica gel, and 1 g sodium sulfate. The lower column (15 mm diameter) is packed in this order: a glass wool plug, 6 g acid alumina, and 1 g sodium sulfate.

11.15.2. Pre-rinse each column with 20 mL hexane, and discard the rinsate.

11.15.3. Put one column above the other.

11.15.4. Add extract to the top column (silica column). Rinse extract vessel 2 times with 1 mL each of hexane and add to column.

11.15.5. Elute 60 mL hexane directly onto acid silica column (upper column).

11.15.6. Discard upper column.

11.15.7. Elute lower column with 10 mL of 20% methylene chloride/hexane. Discard in proper waste stream.

11.15.8. Elute lower column with 30 mL of 65% methylene chloride/hexane. Save and collect in culture tube.

11.15.9. Proceed with additional cleanups as necessary.

11.16. Carbon Column Clean-up (D2 Column)

Prepare an activated Carbon & Silica Gel column as described in below. Refer to the diagram in Figure 3 as well.

11.16.1. Push a glasswool plug down to the 3 inch mark in a pre-cut D2 column.

11.16.2. Add 1 g of 5% activated carbon/silica. Top with a glasswool plug.

11.16.3. With the column oriented with "A" on the top (and the carbon on the lower end of the column), pre-elute with 5 mL 1:1 methylene chloride :cyclohexane.

11.16.4. Discard pre-eluates.

11.16.5. Invert the column so that the column is oriented with the "B" on the top and pre-elute with 3 mL of 1:1 methylene chloride.

11.16.6. Dilute the extract to 1 mL with hexane and transfer to the column (still oriented in the "B" direction).

11.16.7. Rinse sample vial onto the column with 2 x 2 mL 1:1 methylene chloride:cyclohexane.

11.16.8. Elute with 6 mL 1:1 methylene chloride :cyclohexane

11.16.9. Elute with 5 mL 75:25 methylene chloride:methanol

11.16.10. Discard eluates.

11.16.11. Turn the column over (so that the "A" end is on top), and elute with 30 mL of toluene. Collect this eluate.

11.16.12. Concentrate to NEAR dryness using the Rotovap (Section 11.17) or Turbovap (Section 11.18), then proceed to the recovery standard step (Section 11.19).

11.17. Macro-concentration (Rotary Evaporator)

Concentrate the extracts in separate round bottom flasks on rotary evaporator.

11.17.1. Assemble the rotary evaporator according to manufacture's instructions, and warm the water bath. On a daily basis, preclean the rotary evaporator by solvent rinsing. Between samples, 2-3 mL rinses of toluene followed by a 2-3 mL rinse of hexane should be rinsed down the feed tube into a waste beaker.

Rotovap Conditions		
Solvent	**Bath Temperature (C)**	**Vacuum Setting (PSI)**
Toluene	80	25
Hexane	65	15
Methylene Chloride	70	No vacuum applied

11.17.2. Attach the round bottom flask containing the sample extract to the rotary evaporator. Slowly apply vacuum to the system, and begin rotating the sample flask.

11.17.3. Lower the flask into the water bath and adjust the speed of rotation and the temperature as required. At the proper rate of concentration, the flow of solvent into the receiving flask will be steady, but no bumping or visible boiling of the extract will occur.

NOTE: If the rate of concentration is too fast, analyte loss may occur.

11.17.4. For samples requiring % Lipids analysis:

11.17.4.1. Concentrate until the toluene has been completely removed. Add approximately 25 mL hexane and concentrate to ensure that only the lipids are present.

11.17.4.2. Dry the concentration vessel and let stand at room temperature. Weigh the vessel and record on the benchsheet.

11.17.4.3. Calculate % lipids as follows:

$$\%\ \text{Lipids} = \frac{\text{Final Vessel Mass - Initial Vessel Mass}}{\text{Sample Size}} \times 100\%$$

11.17.5. Proceed to extract cleanups, or transfer to a micro concentration vial for the recovery standard step (Section 11.19).

11.18. Micro-concentration (Turbovap)

Concentrate the extracts in 35 mL culture tubes in a turbo-evaporator. The turbo-evaporator model that the laboratory uses can hold up to 50-35 mL culture tubes. Other turbo-evaporator models can be used that may or may not have the same culture tube sizes and/or capacity. Adjust temperature according to solvent (65ºC for toluene and 45ºC for hexane or hexane/ methylene chloride mixtures)

11.18.1. The evaporating times are dependent on sample volume and solvent. The following are examples and can change from sample to sample. Each sample should be checked in intermittent intervals to make sure samples do not go dry.

11.18.2. When evaporating 30 mL toluene, it will normally take approximately 30-50

minutes with the temperature setting described above.

11.18.3. When evaporating 30 mL hexane/ methylene chloride, it will normally take approximately 20-30 minutes with the temperature setting described above.

11.18.4. For samples requiring % Lipids analysis refer to Section 11.17.4.

11.18.5. Proceed to extract cleanups, or transfer to a micro concentration vial for the recovery standard step (Section 11.19).

11.19. Recovery Standard

11.19.1. Transfer extracts to a micro concentration vial (test tubes and other small vessels may also be used)

11.19.2. With a stream of dry, purified nitrogen, reduce the extract volume to approximately 100 µL.

11.19.3. Add 20 µL of the recovery standard solution (Table 2).

11.19.4. With a stream of dry, purified nitrogen, reduce the extract volume to 20 µL.

11.19.5. Transfer the extract to an autoinjection vial and store in the dark at room temperature.

11.19.6. A smaller final volume can be used to decrease the detection limit upon client approval.

11.19.7. A larger final volume can be use to decrease potential matrix interferences, if the column and acid cleanups were unsuccessful.

11.20. Sample Dilution Procedure

11.20.1. Simple dilutions: Dilutions from 2X to 50X can be achieved without respiking the final extract. The calculation to determine the final extract concentration is as follows:

$$\text{Final Conc. of Extract} = \frac{(\text{Conc. of original extract}) \text{ x } (\text{Amount of aliquot taken})}{(\text{Volume of diluted extract})}$$

Ex:
$$\frac{(10\,\text{g}) \text{ x } (2\,\mu\text{L})}{(20\,\mu\text{L}) \text{ x } (100\,\mu\text{L})} = \frac{1\,\text{g}}{100\,\mu\text{L}} \text{ FV}$$

Record the final sample concentration on the extract label.

11.20.2. Complex dilution requiring respiking of IS and RS:

Dilutions greater than 50x must be done by diluting and respiking the extract with IS and RS. This procedure may require serial dilution to be performed. If this procedure is done, then the sample size must be adjusted to reflect the aliquot taken.

Ex. 100X dilution (original sample with 10 g/20 µL final volume)

Take a 2 µL aliquot (1/10 of original sample) and add 18 µL of solvent keeper. Take a 2 µL aliquot of the dilution (1/100 of the original sample), respike with 1 mL IS and 20 µL RS, reduced to 20 µL FV.

Record the final sample concentration of the extract label.

12. CALCULATIONS/DATA REDUCTION

12.1. Not applicable

13. METHOD PERFORMANCE

It must be documented that all applicable system performance criteria specified were met before analysis of any sample is performed. .

13.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required expertise.

13.2. Method Detection Limit

The laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in SOP WS-QA-0006. MDLs are available in the Quality Assurance Department.

13.3. Initial Demonstration

The laboratory must make an initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest.

13.3.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be less than or equivalent to the LCS samples.

13.3.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. Compare these to the laboratory generated QC Limits.

13.4. If any analyte does not meet the acceptance criteria the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

14. POLLUTION CONTROL

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.1. The use of Roto-vaps and Turbo-vaps rather than Kuderna-Danish reduction allows extraction solvents to be collected and disposed of rather than released to the atmosphere.

14.2. Toluene, which is a less hazardous solvent, has been substituted for benzene as an extraction solvent.

14.3. The use of SoxTherm extraction rather than soxhlet extraction, when appropriate, reduces the volume of solvent used.

14.4. Standards should be prepared in volumes consistent with laboratory use to minimize the volume of expired standards that must be discarded.

14.5. All waste will be disposed of in accordance with Federal, State, and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment.

14.6. Do not allow waste solvent to vent into the hoods. All solvent waste is stored in capped containers unless they are being filled.

14.7. Transfer waste solvent from collection cups (tri-pour and similar containers) to jugs and/or carboys as quickly as possible to minimize evaporation.

15. WASTE MANAGEMENT

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to SOP WS-EHS-0001. The following waste streams are produced when this method is carried out.

15.1. Extracted aqueous/leachate samples contaminated with methylene chloride are collected at the fume hood in a 5-gallon or smaller carboy. If the samples are not at a

neutral pH, add small quantities of sodium bicarbonate to bring the waste to neutral. Stir well. Once neutralized, immediately pour the carboy contents into a blue plastic LLE drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the LLE drum to the waste collection area for shipment.

15.2. Extracted soil samples and thimbles, extracted PUF filters, XAD-2 resin, paper funnel filters, glass wool, sodium sulfate, assorted disposable glassware, fish/crawfish or similar materials, silica gel, alumina, and carbon from column clean-ups, contaminated with various solvents and eluates. Dump the materials into a orange contaminated lab trash bucket. When the bucket is full or at the end of the day, tie the plastic bag liner shut and put the lab trash into the steel collection drum in the H3 closet. When the drum is full or after no more than 75 days, move it to the waste collection area for shipment.

15.3. Flammable solvent and methylene chloride waste generated during glassware and sodium sulfate cleaning. Solvent waste collected during roto-vap/turbo-vap reduction of extracted samples. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel solvent drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

15.4. Assorted flammable solvents and methylene chloride waste generated during quartz fiber filter preparation, PUF adsorbent preparation, XAD-2 resin preparation, PUF/XAD-2 cartridge preparation, glassware rinsing and sodium sulfate pre-rinsing.. Waste solvents and methylene chloride collected during roto-rap/turbo-vap reduction of extracted samples. Collect the waste solvents in tripours during use. Empty the tripours into a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the steel drum in the H3 closet. When the drum is full to between two and six inches of the top, or after no more than 75 days, move the steel drum to the waste collection area for shipment.

15.5. Contaminated sulfuric acid used during extract cleanup. Collect the used sulfuric acid in empty, 2.5-liter, plastic coated jars. When full or after one year, whichever comes first, transfer these jars to the waste collection area for shipment.

15.6. Contaminated distilled water used during extract cleanup. Collect the contaminated water in a 1-liter to 4-liter carboy at the fume hood. When the carboy is full, or at the end of your shift, whichever comes first, empty the carboy into the plastic LLE drum in the H3 closet. When full to between two and six inches of the top, or after no more than 75 days, move the plastic drum to the waste collection area for shipment.

16. REFERENCES/CROSS REFERENCES

16.1. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update IV. Method 8290A Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry February 2007.

16.2. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 8290 Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by high-Resolution Mass Spectrometry September 1994.

16.3. SW846, Test Methods for Evaluating Solid Waste, Third edition, Update III. Method 0023A, Sampling Method for Polychlorinated Dibenzo-p-dioxins and Polychlorinated Dibenzofurans Emissions from Stationary Sources. December 1996.

16.4. Compendium Method TO-9A “Determination of Polychlorinated, Polybrominated, and Brominated, Cholorinated Dibenxo-p-dioxins and Dibenzofurans in Ambient Air”, EPA compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, second edition, January 1997.

16.5. Protocol for the Analysis of 2,3,7,8-TCDD by HRGC/HRMS". J. S. Stanley and T. M. Sack, EPA 600/4-86-004.

16.6. “Safety in Academic Chemistry Laboratories", American Chemical Society Publication, Committee on Chemical Safety (3rd Edition, 1979.)

16.7. “Carcinogens - Working with Carcinogens". Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control. National Institute for Occupational Safety and Health. Publication No. 77-206, August 1977.

16.8. "OSHA Safety and Health Standards, General Industry", (29 CFR 1910) Occupational Safety and Health Administration, OSHA 2206 (revised January 1976).

17. METHOD MODIFICATIONS

17.1. Deviations from EPA 8290 and 8290A.

17.1.1. Tetradecane instead of nonane is used as the final solvent to increase the stability of extracts and standards. Tetradecane is less volatile than nonane. Loss of analyte as a result of solvent incompatibility is monitored through recovery checks and calibration acceptance criteria.

17.1.2. Extract clean-ups are performed at the discretion of the analyst when interferences are observed. Then, the analyst should select the clean-up procedure appropriate to the interferent.

17.1.3. Section 7.4.6.4 of Method 8290 indicates that extracts should be transferred with hexane, then toluene. Toluene is used to transfer extracts to maintain compound solubility and minimize analyte loss.

17.1.4. Section 7.5.1.2 of Method 8290 specifies that a NaCl solution should be used for partitioning. Instead, the laboratory uses laboratory water only. NaCl is used to break up emulsions that may form. An analyst may use NaCl, NaOH, or any mechanical means to break up an emulsion.

17.1.5. Section 7.5.3 of Method 8290 specifies that hexane is used as a column elution solvent. The laboratory uses cyclohexane to achieve better and more reproducible separation of the target analyte from the interferent.

17.1.6. Carbon columns are packed with silica gel in place of celite. Elution solvents are changed accordingly. (SOP Section 11.4; Method 8290 Section 7.5.3.2, 8290A Section 7.3.6.).

17.2. Modifications from TO-9A method

17.2.1. Quartz Fiber Filters are cleaned by Soxhlet extraction with methylene chloride, not baked at 400 degrees C for 5 hours.

17.2.2. The PUF material may be pre-cleaned with methylene chloride or other appropriate solvent. The PUFs are not reused.

17.2.3. The $^{37}Cl_4$-2,3,7,8-TCDD surrogate is present at varying levels in the calibration curve (0.5-200 pg/ µL).

17.2.4. Samples are extracted with toluene not benzene.

17.2.5. Concentration is performed by rotary evaporation not Kuderna-Danish.

17.2.6. All cleanup procedures are optional and applied based on the analyst's discretion.

17.2.7. The laboratory uses 2 labeled recovery standard for the quantitation of labeled internal standards.

17.2.8. The final volume is adjusted to 20 µL in tetradecane.

17.2.9. Calibration and quantitation are performed in accordance to this SOP.

18. ATTACHMENTS

18.1. Table 1 - Types of Matrices

18.2. Table 2 - Composition of Sample Fortification and Recovery Standard Solutions.

18.3. Table 3 - The Seventeen 2,3,7,8-Substituted PCDD and PCDF Congeners

18.4. Figure 1 - Analysis Flowchart

18.5. Figure 2 — IFB column cleanup

18.6. Figure 3 — D2 Column cleanup

18.7. Appendix A - Periodic Wipe Test Performance

19. REVISION HISTORY

19.1. WS-IDP-0005, Revision 1.5, Effective 12/21/2012

19.1.1. Clarified extraction procedure by revising Section(s) 11.8.1- 11.8.4 and adding an extra extraction step (Section 11.8.3).

19.1.2. Editorial revisions. .

19.2. WS-IDP-0005, Revision 1.4, Effective 03/20/2012

19.2.1. Appended to Section 2.2: “This method can also use solid phase extraction (SPE), however, Test America West Sacramento is in the developmental stages for this extraction type and is not currently certified for its use.”

19.2.2. Editorial changes.

19.3. WS-IDP-0005, Revision 1.3., Effective 06/10/2011

19.3.1. Added Section 11.9: Aqueous Samples (Solid Phase Extraction).

19.3.2. Editorial revisions.

19.4. WS-IDP-0005, Revision 1.2, Effective 2/11/2011

19.4.1. Added benzene to Section 5.2 Table..

19.4.2. Editorial revisions.

19.5. WS-IDP-0005, Revision 1.1, Effective 2/12/2010

19.5.1. Section 11.2 – updated SOP reference from SAC-ID-0009 to WS-ID-0009.

19.5.2. Section 11.6.1 – changed: “Prior to loading samples, run the system through

a cleaning cycle (approximately 3 hours)" to "(approximately 1 hour)."

19.5.3. Section 11.6.8 – changed "…fresh charge (140 mL) of toluene…" to "…fresh charge (150 mL) of toluene….".

19.5.4. Section 11.16.1 – inserted in Table "No vacuum applied" under vacuum setting (PSI) for solvent Methylene chloride.

19.6. WS-IDP-0005, Revision 1, Effective 10/2/2008

19.6.1. Added 8290A references.

19.6.1.1. Extract and standard storage.

19.6.1.2. Removal of MS/MSD.

19.6.2. Updated to TestAmerica format.

19.6.3. Separated the analytical steps from the preparation steps, this SOP is concerned only with the sample preparation.

19.7. WS-ID-0005, Revision 6.7, Effective 8/21/2008

19.7.1. Changed the word "toluene" to "acetone" in 7.11.2.

19.8. WS-ID-0005, Revision 6.6, Effective 4/9/2008

19.8.1. Added South Carolina rule to prepare an MS/MSD with every batch.

19.8.2. Modified to include extraction and analysis of ambient air samples collected in filter/PUF material.

TABLE 1

Types of Matrices, Sample Sizes and 2,3,7,8-TCDD-Based Method Calibration Limits (Parts per Trillion)

	Water	Soil Sediment Paper Pulp	Fly Ash	Human/ Fish Tissue	Adipose Tissue	Sludges, Fuel Oil	Still-Bottom	Ambient or Source Samples
Lower MCL(a)	0.01	1.0	2.0	1.0	2.0	10	20	40
Upper MCL(a)	4.0	400	400	400	400	2000	4000	8000
Weight (g)	1000	10	10	10	10	2.0	1.0	1 sample
IS Spiking Levels (ng)	2.0	2.0	2.0	2.0	2.0	2.0	2.0	4.0
Final Extract Volume (μL)	20	20	20	20	20	20	20	20

(a) For other congeners, multiply the values by 1 for TCDF, by 5 for PeCDD/PeCDF/HxCDD/HxCDF/HpCDD/HpCDF, and by 10 for OCDD/OCDF.

TABLE 2

Composition of the Sample Fortification and Recovery Standard Solutions

Analyte	Semple Fortification Solution Concentration pg/µL; Solvent: Isooctane	Recovery Standard Solution Concentration pg/µL; Solvent: Tetradecane
$^{13}C_{12}$ -2,3,7,8-TCDD	2[a], 100[c]	--
$^{13}C_{12}$ -2,3,7,8-TCDF	2[a], 100[c]	--
$^{13}C_{12}$ -1,2,3,4-TCDD	--	100
$^{13}C_{12}$ -1,2,3,7,8-PeCDD	2[a], 100[c]	--
$^{113}C_{12}$ -1,2,3,7,8-PeCDF	2[a], 100[c]	--
$^{13}C_{12}$ -1,2,3,6,7,8-HxCDD	2[a], 100[c]	--
$^{13}C_{12}$ -1,2,3,4,7,8-HxCDF[d]	2[a], 100[c]	--
$^{113}C_{12}$ -1,2,3,7,8,9-HxCDD	--	100
$^{13}C_{12}$-2,3,7,8-TCDD[b][c]	0.8[b],100[c]	
	100[c]	
$^{13}C_{12}$ -2,3,4,7,8-PeCDF[c]	100[c]	
$^{13}C_{12}$ -1,2,3,6,7,8-HxCDF[c][d]	100[c]	
$^{13}C_{12}$ -1,2,3,4,7,8-HxCDD[c]	100[c]	
$^{13}C_{12}$ -1,2,3,4,7,8,9-HpCDD[c]	100[c]	
$^{13}C_{12}$ -1,2,3,4,6,7,8-HpCDD	2[a], 100[c]	--
$^{13}C_{12}$ -1,2,3,4,6,7,8-HpCDF	2[a], 100[c]	--
$^{13}C_{12}$ -OCDD	4[a], 200[c]	--

(a) Standard 8290, Method 23, Method 0023A, TO9 and TO9A Sample Fortification Solution concentrations
(b) Method TO9 and TO9A surrogate concentrations
(c) Method 23 and Method 0023A surrogate concentrations
(d) $^{13}C_{12}$ -1,2,3,6,7,8-HxCDF is used as a Sample Fortification Solution and $^{13}C_{12}$-1,2,3,4,7,8-HxCDF is used as a surrogate solution in Method 23 and Method 0023A

TABLE 3

The Seventeen 2,3,7,8-Substituted PCDD and PCDF Congeners

PCDD	PCDF
2,3,7,8-TCDD(*)	2,3,7,8-TCDF(*)
1,2,3,7,8-PeCDD(*)	1,2,3,7,8-PeCDD(*)
1,2,3,6,7,8-HxCDD(*)	2,3,4,7,8-PeCDF
1,2,3,4,7,8-HxCDD	1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDD(+)	1,2,3,7,8,9-HxCDF
1,2,3,4,6,7,8-HpCDD(*)	1,2,3,4,7,8-HxCDF(*)
1,2,3,4,5,6,7,8-OCDD(*)	2,3,4,6,7,8-HxCDF
	1,2,3,4,6,7,8-HpCDF(*)
	1,2,3,4,7,8,9-HpCDF
	1,2,3,4,5,6,7,8-OCDF

(*)The ^{13}C -labeled analog is used as an internal standard.
(+)The ^{13}C -labeled analog is used as a recovery standard.

FIGURE 1 — Flowchart of Process

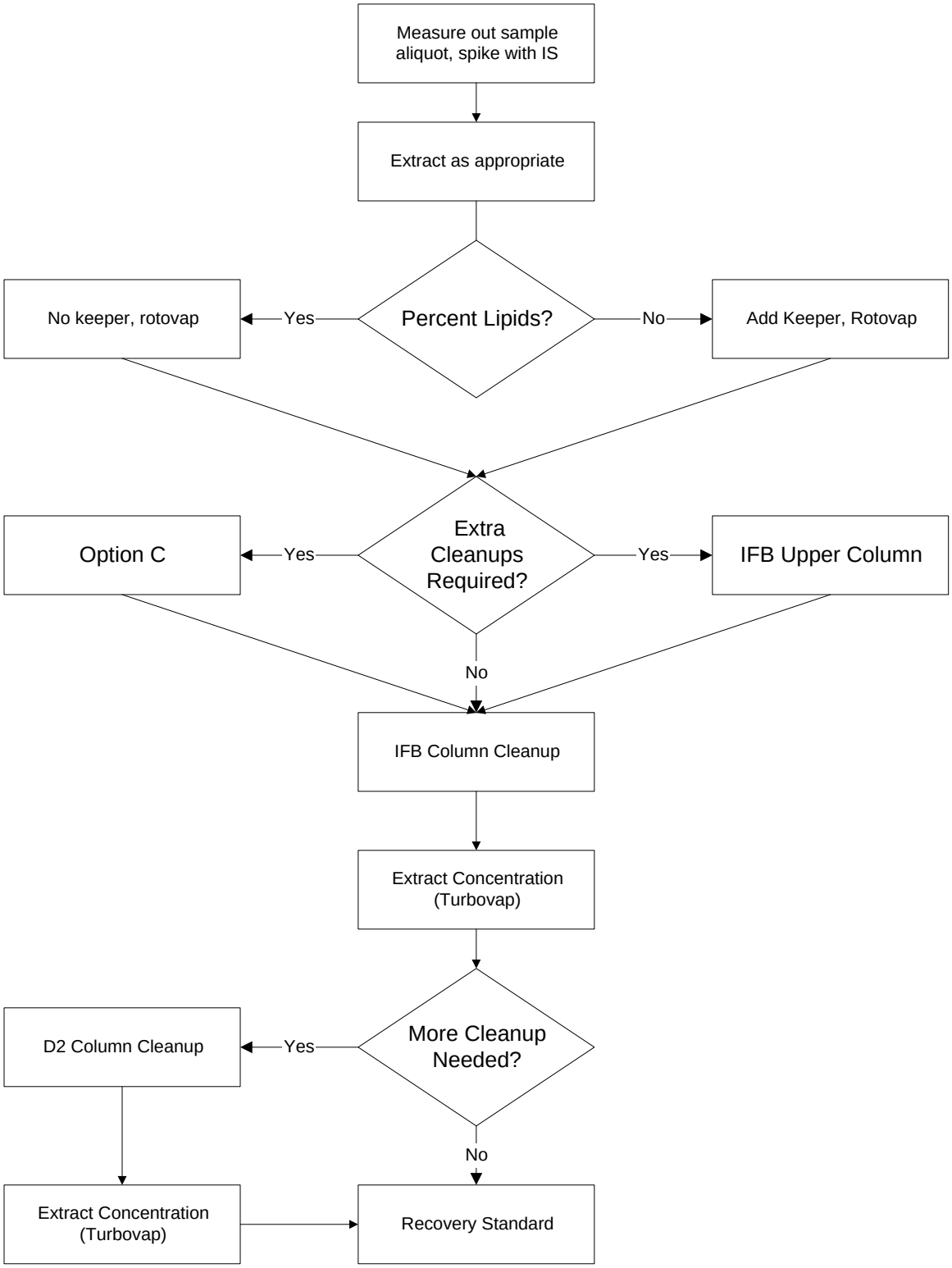


Figure 2 – Diagram of IFB Column Cleanup

Use 20 mm column for top column (IFB Column)
Use 16 mm column for bottom column* (Acid Alumina)
Note: Upper and lower columns are piggy backed for IFB cleanup, upper column only can be used for additional cleaning.

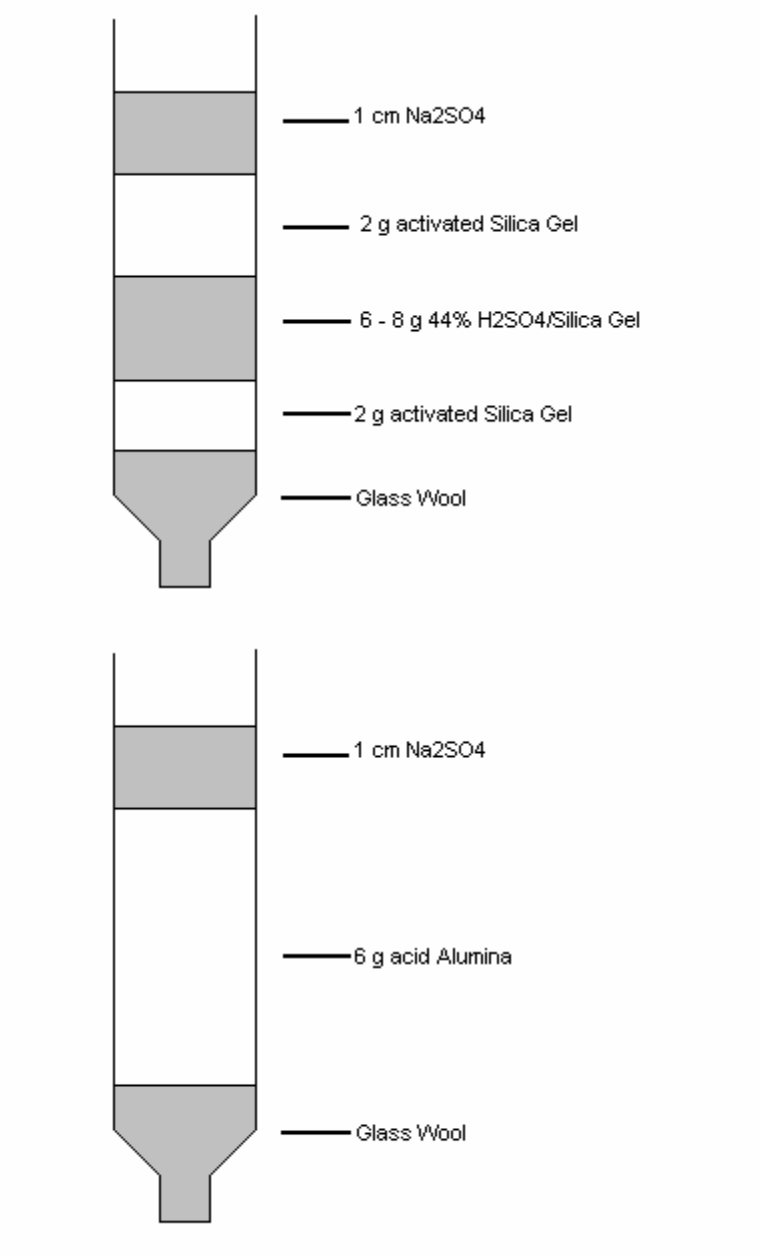
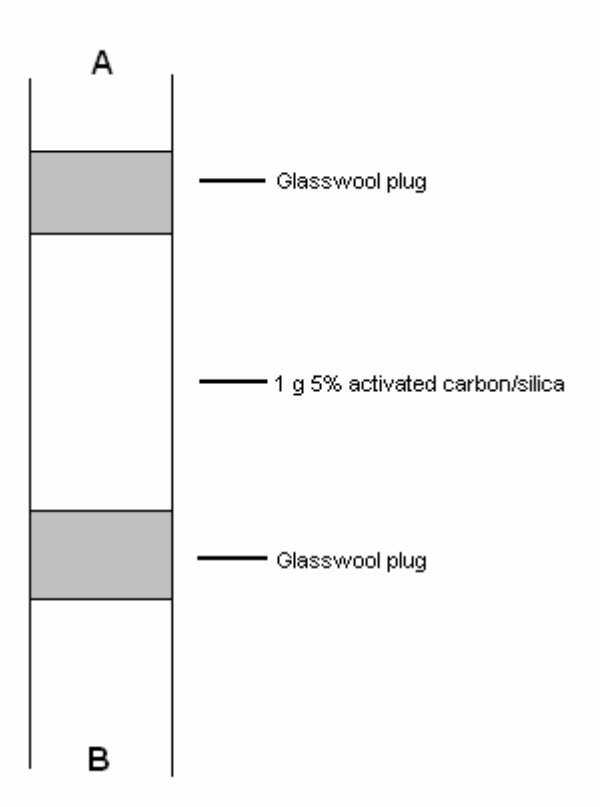


Figure 3— D2 Carbon Column:



APPENDIX A — Screening the Laboratory for 2,3,7,8 Congeners

This procedure is designed for the periodic evaluation of potential contamination by 2,3,7,8-substituted PCDD/PCDF congeners of the working areas inside the laboratory.

PERFORMING WIPE TEST
Perform the wipe tests on surface areas of two inches by one foot with laboratory wipers saturated with distilled-in-glass acetone or appropriate solvent using a pair of clean stainless steel forceps. Use one wiper for each of the designated areas. Combine the wipers to one composite sample in an extraction jar containing 200 mL distilled-in-glass hexane. Place an equal number of unused wipers in 200 mL hexane and use this as a control.

SAMPLE PREPARATION
Close the jar containing the wipes and 200 mL hexane and extract for 20 minutes using a wrist-action shaker. Use an appropriate means to reduce the volume to approximately 1.0 mL. Put through an alumina column to clean up potential interfering compounds. Add appropriate amount of recovery standard.

EXTRACT ANALYSIS
Concentrate the contents of the vial to a final volume of 20 µL (either in a minivial or in a capillary tube). Inject 2 µL of each extract (wipe and control) onto a capillary column and analyze for 2,3,7,8-substituted PCDDs/PCDFs as specified in the analytical method Section 11 (this exhibit). Perform calculations according to Section 12 (this exhibit).

REPORTING FORMAT
Report the presence of 2,3,7,8-substituted PCDDs and PCDFs as a quantity (pg or ng) per wipe test experiment (WTE). Under the conditions outlined in this analytical protocol, a lower limit of calibration of 25 pg/WTE is expected for 2,3,7,8-TCDD. A positive response for the blank (control) is defined as a signal in the TCDD retention time window at any of the masses monitored which is equivalent to or above 8 pg of 2,3,7,8-TCDD per WTE. For other congeners, use the multiplication factors listed in Table 1, footnote (a) (e.g., for OCDD, the lower MCL is 25 x 5 = 125 pg/WTE and the positive response for the blank would be 8 x 5 = 40 pg). Also, report the recoveries of the internal standards during the simplified cleanup procedure.

FREQUENCY OF WIPE TESTS
Wipe tests should be performed when there is evidence of contamination in the method blanks.

CORRECTIVE ACTION
An upper limit of 25 pg per TCDD isomer and per wipe test experiment is allowed. (Use multiplication factors listed in footnote (a) from Table 1 for other congeners.) This value corresponds to the lower calibration limit of the analytical method. Steps to correct the contamination must be taken whenever these levels are exceeded. To that effect, first vacuum the working places (hoods, benches, sink) using a vacuum cleaner equipped with a high-efficiency

particulate absorbent (HEPA) filter and then wash with a detergent. A new set of wipes should be analyzed before anyone is allowed to work in the dioxin area of the laboratory.

The test results and the decontamination procedure must be reviewed with EH&S.

APPENDIX F

WELL INFORMATION

GZA GEOENVIRONMENTAL, INC.
140 BROADWAY, PROVIDENCE, RHODE ISLAND
GEOTECHNICAL/GEOHYDROLOGICAL CONSULTANTS

PROJECT
DEFENSE FUEL SUPPORT POINT
TANK FARM 3 PORTSMOUTH, RI

REPORT OF BORING No. GZ-301
SHEET 1 OF 1
FILE No. 31288.3
CHKD. BY ABU

BORING Co. GZA DRILLING, INC.
FOREMAN CHRIS LENLING
GZA ENGINEER MARK DALPE

BORING LOCATION SEE LOCATION PLAN
GROUND SURFACE ELEVATION DATUM
DATE START 10/31/94 DATE END 10/31/94

SAMPLER: UNLESS OTHERWISE NOTED, SAMPLER CONSISTS OF A 2" SPLIT SPOON DRIVEN USING A 140 lb. HAMMER FALLING 30 In.

CASING: UNLESS OTHERWISE NOTED, CASING DRIVEN USING A 300 lb. HAMMER FALLING 24 In.

CASING SIZE: HSA 3 3/4" ID OTHER:

GROUNDWATER READINGS

DATE	TIME	WATER	CASING	STABILIZATION TIME
11-1-94	0830	DRY	OW	19 HOURS
11-4-94	0830	DRY	OW	4 DAYS
11-21-94	1210	18.7	OW	20 DAYS
12-15-94	1430	13.7	OW	6 WEEKS

DEPTH	CASING	BLOWS	SAMPLE No.	SAMPLE PEN./REC.	SAMPLE DEPTH (Ft.)	SAMPLE BLOWS/6"	SAMPLE DESCRIPTION (Burmister CLASSIFICATION)	STRATUM DESCRIPTN	EQUIPMENT INSTALLED	FIELD TESTING PID	FID	ODOR	REMKS
			S-1	24/16	0.2-2.2	7-10	Medium dense, brown, coarse to fine+ SAND, some +Silt, some Gravel			0.6	4.2	SL	1 2 3
						14-10		SILTY GRAVELLY SAND					
5			S-2	24/6	5-7	8-27	Very dense, gray, coarse to fine+ SAND and Gravel, little+ Silt						
						37-30			6'				
							APPARENT TOP OF BEDROCK	8'	7'	ND	ND	N	
10			S-3	20/20	10-11.7	5-16	Weathered SHALE	WEATHERED SHALE		ND	ND	N	
						50-100/2"							
15													
20							End of Exploration at 20'+						4
25													
30													
35													

EQUIPMENT INSTALLED: PVC RISER; Grout; 6' Bent. Seal; 7' Filter Sand; 9.5' PVC SCREEN; 19.5'

REMARKS:
1. Soil head space screening was performed employing a Century Systems Model OVA-128 equipped with a hydrogen flame ionization detector (FID), and a HNU System model PI-101 photoionization detector (PID) equipped with a 10.2 eV lamp. Readings are in parts per million (ppm). ND indicates not detected above instrument detection limit of 0.1 ppm.
2. N = no odor, SL=slight odor
3. Slight petroleum-like odor noted from 0-2'+.
4. 10' of .02" slotted, 2.0" diameter, Sch 40, PVC wellscreen was placed from 19.5' up to 9.5'+ and topped with 9.5'+ of solid PVC riser tube. Filter sand was poured up to 7'+ and a bentonite seal placed from 7' up to 6'+. Bentonite/cement grout was placed from 6' up to the surface. The well head was secured at grade with a flush mounted, 1' long, aluminum curb box grouted in place. (surface seal: 2'x2'x4" concrete pad).

NOTES:
1) STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL.
2) WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER MAY OCCUR DUE TO OTHER FACTORS THAN THOSE PRESENT AT THE TIME MEASUREMENTS WERE MADE

GZA

BORING No. GZ-301

GZA GEOENVIRONMENTAL, INC.
140 BROADWAY, PROVIDENCE, RHODE ISLAND
GEOTECHNICAL/GEOHYDROLOGICAL CONSULTANTS

PROJECT
DEFENSE FUEL SUPPORT POINT
TANK FARM 3 PORTSMOUTH, RI

REPORT OF BORING No. GZ-314
SHEET 1 OF 1
FILE No. 31288.3
CHKD. BY ABU

BORING Co. GZA DRILLING, INC.
FOREMAN CHRIS LENLING
GZA ENGINEER MARK DALPE

BORING LOCATION SEE LOCATION PLAN
GROUND SURFACE ELEVATION DATUM
DATE START 11/01/94 DATE END 11/02/94

SAMPLER: UNLESS OTHERWISE NOTED, SAMPLER CONSISTS OF A 2" SPLIT SPOON DRIVEN USING A 140 lb. HAMMER FALLING 30 In.

CASING: UNLESS OTHERWISE NOTED, CASING DRIVEN USING A 300 lb. HAMMER FALLING 24 In.

CASING SIZE: HSA 3 3/4" ID OTHER:

GROUNDWATER READINGS

DATE	TIME	WATER	CASING	STABILIZATION TIME
11-3-94	0730	26.4	OW	18 HOURS
11-21-94		26.1	OW	2 WEEKS

DEPTH	CASNG	BLOWS	SAMPLE No.	PEN./REC.	DEPTH (Ft.)	BLOWS/6"	SAMPLE DESCRIPTION (Burmister CLASSIFICATION)	STRATUM DESCRIPTN	EQUIPMENT INSTALLED	FIELD TESTING PID	FID	ODOR	REMKS
			S-1	24/20	0-2	4-8	Medium dense, brown/gray, coarse to fine+ SAND, little+ Gravel, little Silt	SILTY GRAVELLY SAND	PVC RISER	ND	ND	N	1 2
						14-22		3' SILTY SAND	Grout				
5			S-2	24/7	5-7	6-12	Medium dense, tan, fine SAND, some Silt		6' Bent. Seal	ND	ND	N	
						14-18		7' SILTY GRAVELLY SAND	7'				
10			S-3	24/19	10-12	14-22	Very dense, gray, coarse to fine+ SAND and Gravel, little + Silt		9.5'	ND	ND	N	
						38-48		12'	PVC Screen				
							APPARENT TOP OF BEDROCK						
15			S-4	1<1	15-15.1	100/1"	REFUSAL: SHALE		Filter Sand	ND	ND	N	
20													
25								SHALE					
30													
35													
40							END OF EXPLORATION AT 40'		39.5'				3 4

REMARKS: 1. Soil head space screening was performed employing a Century Systems Model OVA-128 equipped with a hydrogen flame ionization detector (FID), and a HNU System model PI-101 photoionization detector (PID) equipped with a 10.2 eV lamp. Readings are in parts per million (ppm). ND indicates not detected above instrument detection limit of 0.1 ppm. 2. N = no odor. 3. No visual/olfactory indications of contamination noted.
4. 30' of .02" slotted 2.0" diameter, Sch 40, PVC wellscreen was placed from 39.5' up to 9.5'+ and topped with 9.5'+ of solid PVC riser tube. Filter sand was poured up to 7'+ and a bentonite seal placed from 7' up to 6'+. Bentonite/cement grout was placed from 6' up to the surface. The wellhead was secured with a flush mounted 1' long aluminum curb box grouted into the surface (surface seal: 2'x2'x4" concrete pad).

NOTES: 1) STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL.
2) WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER MAY OCCUR DUE TO OTHER FACTORS THAN THOSE PRESENT AT THE TIME MEASUREMENTS WERE MADE

GZA

BORING No. GZ-314

GZA GEOENVIRONMENTAL, INC.
140 BROADWAY, PROVIDENCE, RHODE ISLAND
GEOTECHNICAL/GEOHYDROLOGICAL CONSULTANTS

PROJECT
DEFENSE FUEL SUPPLY CENTER
TANK FARM 3, PORTSMOUTH, RI

REPORT OF BORING No. GZ-318
SHEET 1 OF 1
FILE No. 31288.9
CHKD. BY ABO

BORING Co. GZA DRILLING, INC.
FOREMAN CHRIS LENLING
GZA ENGINEER MARK DALPE

BORING LOCATION REFER TO EXPLORATION LOCATION PLAN
GROUND SURFACE ELEVATION ____ DATUM ____
DATE START 11/14/95 DATE END 11/15/95

SAMPLER: UNLESS OTHERWISE NOTED, SAMPLER CONSISTS OF A 2" SPLIT SPOON DRIVEN USING A 140 lb. HAMMER FALLING 30 In.

CASING: UNLESS OTHERWISE NOTED, CASING DRIVEN USING A 300 lb. HAMMER FALLING 24 In.

CASING SIZE: 3 3/4" HSA TO 18'+ OTHER: NX TYPE ROCK CORE COLLECTED FROM 19' - 29'+

GROUNDWATER READINGS

DATE	TIME	WATER	CASING	STABILIZATION TIME
11-15-95	0740	14.8	19'	17.5 HOURS
11-16-95	0730	15.2	WELL	21 HOURS
11-27-95	0915	15.1	WELL	12 DAYS

DEPTH	CASING BLOWS	SAMPLE No.	SAMPLE PEN./REC.	SAMPLE DEPTH (Ft.)	SAMPLE BLOWS/6"	SAMPLE DESCRIPTION	STRATUM DESCRIPTION	EQUIPMENT INSTALLED	FIELD TESTING PID	FID	ODOR	REMKS
0		S-1	24/19	0-2	12-14	Medium dense, brown, TOPSOIL changing at 0.5'+ to tan/gray, coarse to fine+ SAND and Gravel (FILL)	TOPSOIL	.8' PVC RISER; BENT. GROUT	4.0	22	N	1 2
					8-6		0.5' RE-WORKED TILL (FILL)					
							3' TILL					
5		S-2	24/18	5-7	18-25	Very dense, gray/orange-brown, coarse to fine+ SAND and Gravel, little+ Silt (TILL)		5' BENT. SEAL; 6'	ND	0.4	N	
					30-27							
						APPARENT TOP OF BEDROCK		8'				
10							8.5' WEATHERED SHALE	FILTER SAND				
15								PVC SCREEN				3
	CORE/TIME MIN./FT.											
20	1.5	C-1 R-1	60/58	19-24	RQD=35%	Soft, moderately weathered, moderately fractured, (horizontal to 60° fracture angles) with iron stained fracture surfaces, light gray, slightly metamorphosed SHALE. Two approximate 3" layers of slightly metamorphosed SILTSTONE	19' MODERATELY WEATHERED SHALE					
	1.3				REC=97%							
	1.5											
	1.5											
	1.0											
25	1.0	C-14 R-2	60/48	24-29	RQD=67% REC=80%	Soft, fresh, very slightly fractured (40° fracture angles) with iron stained fracture surfaces, light gray slightly metamorphosed SHALE.	24' FRESH SHALE	28'+				4 5
	1.0											
	0.8											
	0.8											
	1.2											
30						END OF EXPLORATION AT 29'+						
35												

1. Soil headspace screening was conducted employing a HNU systems Model PI-101 photoionization detector (PID) equipped with a 10.2 eV lamp and a Century Model OVA-128 equipped with a hydrogen flame ionization detector (FID). Readings are reported in parts per million (ppm). ND indicates not detected above the instrument detection limit of 0.1 ppm.
2. N indicates no odor.
3. Strong petroleum-like odor noted in SHALE cuttings (15-18'+); FID readings of >10 ppm were containerized (approximate 15 gallon volume of rock cuttings).
4. 20' of .02" slotted, 2.0" diameter, Sch 40 PVC well screen was placed from 28'+ up to 8'+ and topped with 8' of solid PVC riser tube. Filter sand was poured up to 6'+ and a bentonite seal (hydrated upon placement) placed from 6' up to 5'+. Bentonite grout was placed from 5' up to 0.8'+. The wellhead was secured with a 0.8' long flush mounted aluminum curb box grouted into the surface with concrete (2'x2'x4" thick concrete).
5. Approximately 80 gallons of drill water remained in the formation upon well completion.

GZA